POROSITY ANALYSIS IN STARCH IMBUED HANDSHEETS CHALLENGES USING IMPULSE DRYING AND METHODS FOR IMAGE ANALYSIS

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POROSITY ANALYSIS IN STARCH IMBUED HANDSHEETS CHALLENGES USING IMPULSE DRYING AND METHODS FOR IMAGE ANALYSIS

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Dedicated to Mad, for her loving support and encouragements in almost 3 years
To my parents, for supporting me without a clue of what I am doing these last years
To Dr. Orloff who has been a mentor to me and an enlightening advisor
To Barbara, for her support as well as her kindness to believe in me
And To Chris & Sowatha, who probably don't know that they own a place here,
As well as Lander (T'y es preque!)
The others, you know who you are … The list would be too long
(Et Madahine n'est pas oubliée non plus!)

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Å	Angstrom

Abbreviations

BTU	British Thermal Unit
BW	Basis Weight
CD	Cross Direction
CLAHE	Contrast-limited adaptive histogram equalization
CSF	Canadian Standard Freeness
DW	Dry Weight (either air or oven dried)
FFT	Fast Fourier Transform
I.P.S.T.	Institute of Paper Science and Technology
LoG	Laplacian of a Gaussian
MD	Machine Direction
M.T.S.	Electrohydraulic press
ODE	Ordinary Differential Equation
RGB	Red-Green-Blue
S.E.M.	Scanning Electron Microscope
TAPPI	Technical Association of the Pulp and Paper Industry
TIFF	Tagged Image File Format

SUMMARY

In about 30 years of experiments and development, impulse drying is now considered as a well known technology and a good candidate in the constant effort to save energy in the paper industry. The drying section is indeed the most expensive section in the process of paper production. However, this potential technology has a major disadvantage, stopping its implementation in the industry. Paper, which is a porous material with a variable compressibility, experienced a sudden release of energy at the nip opening during impulse drying. Under these conditions of high intensity process (both in temperature and pressure), the fiber mat has a tendency to delaminate. This web disruption is a critical issue against impulse drying.

This thesis comes up with a new approach to the problem. These last years, the technology itself has been addressed in this issue and many improvements have been reached in terms of energy release (heat transfer control, material coating…). The novel idea is then to investigate the inner structure of the paper once it has been coated with starch to a large extent (up to 10 or 20% of the relative basis weight). Starch is known for its large use in industry, but also its capability to expand under high temperature. Hence, both relative strength and bulking effects are investigated in this thesis, using numerous experiments with variable temperatures and pressures, along with ultrasonic testing and image analysis. We have the opportunity to appreciate the phenomenon of heat transfer and mass transport in the coated medium, while reaching promising results in terms of strength and bulk. These are finally investigated using scanning electron microscopy as a first step toward a pore expansion model for starch imbued handsheets.

CHAPTER 1

INTRODUCTION

As anybody familiar with the paper industry knows, the drying section in the paper process is one of the most expensive both in terms of capital and operation costs. Improving this section is therefore a goal consistently relevant as it is clear that expenses in the paper industry won't decrease in the future (TAPPI conference – Kingsport 2007). This drying section is still based on the extensive use of steam heated rolls organized in various configurations depending on the facilities and the choice of paper grade for the production. However, a normal configuration can use more than 40 of these gigantic steam filled rolls to facilitate the water evaporation in the paper web. The use of steam, among other problems, impacts the performance of such process by the simple fact that it offers a significant thermal resistance inside each roll. Improving such a section would clearly have a great impact on the economy of the paper industry. Two candidates are well known now for this purpose. These two processes have been fairly well studied in the last 20 to 30 years: namely press drying and impulse drying. Besides the fact that these two processes use high intensity performances (high range of temperatures and pressures) the main difference is the nip residence time. Press drying operates on a long nip residence time which can go up to 5 seconds, while impulse drying is a very fast process whose nip residence time ranges between 20 to 40 milliseconds.

This thesis is dedicated, but not exclusive, to impulse drying. The physics behind the process of impulse drying is well known nowadays. To summarize, the impulse drying process may be broken down to various intervals. Impulse drying is classical wet pressing followed by a two-phase flow/flash evaporation process. In details, the first time interval is a wet pressing process, the second the start-up of a nucleate pool boiling process with the creation of a vapor front, the third one a process of flash evaporation

concerning the bound water and finally an unrestrained continued flash evaporation process at the nip opening. While these heat and mass transport processes are very efficient, they also have the disadvantage to trigger delamination of paper at the nip opening. In other words, the paper web experiences a sudden disruption which is clearly undesirable.

This issue among others has delayed the use of impulse drying on an industry scale. Attempts to resolve delamination have been focused on a technological level, and efforts have been put in the study of such delamination conditions. Some partial solutions came through the observation that the heat transfer rate and also the external pressure conditions were all playing a role on the sudden excessive energy release experienced by the web at the nip opening. Hence, a technological solution included the use of “low thermal” mass materials as a coating layer on the heated roll used in impulse drying. However, it’s been observed that by doing so, internal bulking was totally while it had been counted as one of the advantages of impulse drying. So the challenge is to avoid delamination while allowing bulking of the web.

These preliminaries experiments and observation have lead to the idea that the parameter to play with should be the paper web instead of the technology itself. Reasoning that starch is a commonly used product in the paper industry, both as a strengthening and retaining agent, it could be of some use in our problem. Another interesting advantage of starch is its behavior under a certain level of temperature, namely its capacity for expansion. We have here the necessary tools to start our study.

As a first approach, it has been decided that various starches should be studied in order to determine the optimum choice, in various conditions of pressure and temperature. Following this first set of experiments, it’s been indeed shown that a fairly good level of strength, along with bulking effect, could be attained with an amount of starch somewhere between 10 and 20% of the total mass of a designated sample.

Under these conditions, a series of ultrasonic testing was performed, while a hypothesis of work was formulated. It was believed that using starch to this extent of proportion serves the paper web by coating the fibers, hence reinforcing this porous structure. Acting on the porous structure in this manner, and knowing the capability of starch to expand under high temperatures, it's been hypothesized that the inner structure of the fiber mat was influenced both through its porosity and pore connectivity. In other words, it is believed that the porous structure would be closed and would exhibit less connectivity, at least in terms of micropores. If it's the case, then flash evaporation during impulse drying has a great chance to be contained, while a high level of internal pressure increases the bulk of the paper web. While it could be an issue in a normal fiber matrix, the fact that starch is also a strengthening agent seems to serve our purpose of avoiding delamination fairly well.

This offers both an exciting opportunity for this technology, and an interesting challenge in understanding the underlying physics. It is therefore been decided that a complete study should be undertaken. Hence, a series of experiments was formulated in order to investigate first the impact of temperature on starch imbued handsheets during impulse drying, along with ultrasonic testing; and then the influence of the pressure gradient at the nip opening, as it proves to be a critical factor in the processes going on inside the porous structure during high intensity processes such as impulse drying. In parallel, since we were experimenting on the porous structure, it seems natural to investigate this structure along with its critical parameters such as porosity, pore-size distribution and sheet permeability. All these were indeed critical when dealing with two-phase flow in a porous media, and essential in determining the pore connectivity of the fiber mat reinforced by starch. Keeping in mind that we are influencing in this manner, indirectly, the venting capability of the paper web, it is important to keep track of the structure. With porous structure study, it is essential to be as accurate as possible, hence

the use of scanning electron microscopy along with image analysis program testing. This is indeed a big part in the context of this thesis. Approaching the problem of delamination on the point of view of porous structure has given a good insight on the impact of our solution. We have been able to determine that replacing a fair amount of fibers by this same amount of starch, we were impacting the paper web during impulse drying by controlling the problem of delamination. This modifies the porous structure by creating more macropores than usually encountered in a normal paper web.

CHAPTER 2

IMPULSE DRYING

In the paper industry, more than ever, the question of energy saving is a critical parameter along the overall process of paper production. Recent reviews emphasize the fact the energy cost is not expected to decrease, and it's now part of the commercial strategy to study all the industry components in order to improve this sector.
One section in the paper industry that still needs improvements on this matter is the drying section. On this concern, impulse drying is still a promising technology, which has been studied for about 30 years now.

Conventional Press Drying

A quick overview of drying technologies is necessary to understand the reasons leading to the study of impulse drying. In this section, a brief analysis of current dry end operations is given, along with key parameters of dryer section.

General Description and Performance

(*In the following, please refer to reference [1] for more information*)

Conventional paper machines are using various configurations of a series of large diameter, rotating, and steam-filled cylinders. The configurations depend mainly on the grade produced.

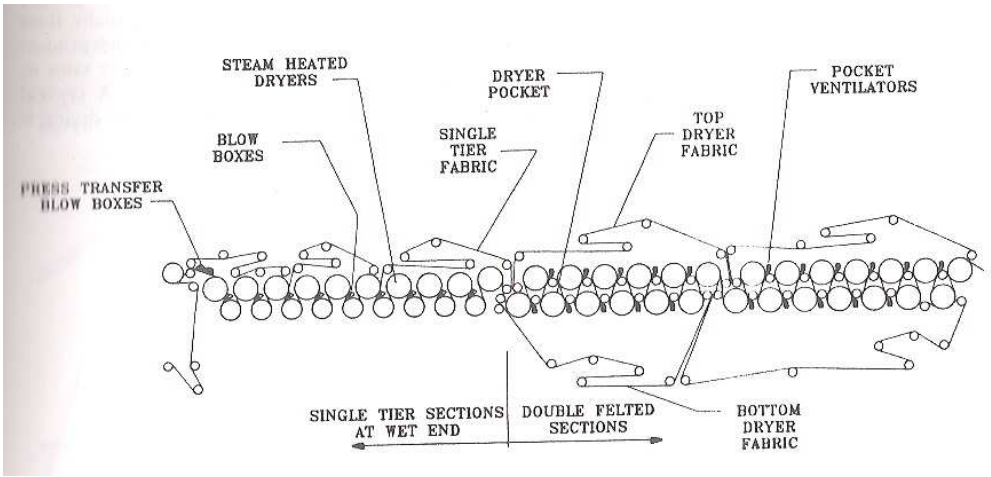


Illustration 1: Typical dryer configuration for lightweight papers

The massive dryer section accounts for the most expensive part of a paper machine in terms of capital cost, as well as the most costly to operate regarding its high energy consumption.

It is easier understood when assessing the performance of a dryer section. Two parameters are essential to characterize it: one is the evaporation drying rate; the other is the steam economy. Evaporation drying rate is measured as pounds of water evaporated per hour per square foot of dryer surface contacted. Although a high evaporation rate is desired, this cannot be achieved without considering constraints defined by the type of product handled (furnish, paper grade) and the desired product quality. One major technological constraint is that this rate is greatly influenced by the steam pressure used inside the drying cylinders.

Steam economy is measure as thousands of BTU's per pound of water evaporated (or kJ per kg). In other words, it corresponds to the mass of steam necessary to evaporate a unit mass of water. For the most economical operation, a low steam usage is desired. Well designed mill with well maintained system has usually a value of 1.3 kg of steam per kg of water evaporated. However, as a recent conference presentation has shown, this ideal situation is far from being reached by most companies in the US (TAPPI Fall Conference

2007 – Kingsport) and great efforts are still to be made in terms of energy savings, particularly in the drying section, especially on steam/condensate and air handling systems.

It is clear under these considerations that a complete different approach to dry end operations combining energy efficiency and low cost operations is desirable on many levels in the paper industry.

Drying process

To further specify the approach, it is also essential to understand the drying process as handled by most companies so far. Heat energy is provided to the wet web by steam as it condenses inside the drying cylinder. Latent heat is extracted at saturation temperature. As said above, it depends strongly on the steam pressure inside the cylinders. The fact that this condensate process occurs is also a limitating parameter to the drying process. Thermal resistance is building as a condensate layer forms inside the cylinder, another resistance is offered as well by the air layer between sheet and cylinder. If non-condensibles are allowed to accumulate within the steam cylinders, they can adversely affect heat transfer and can also cause non uniform drying.

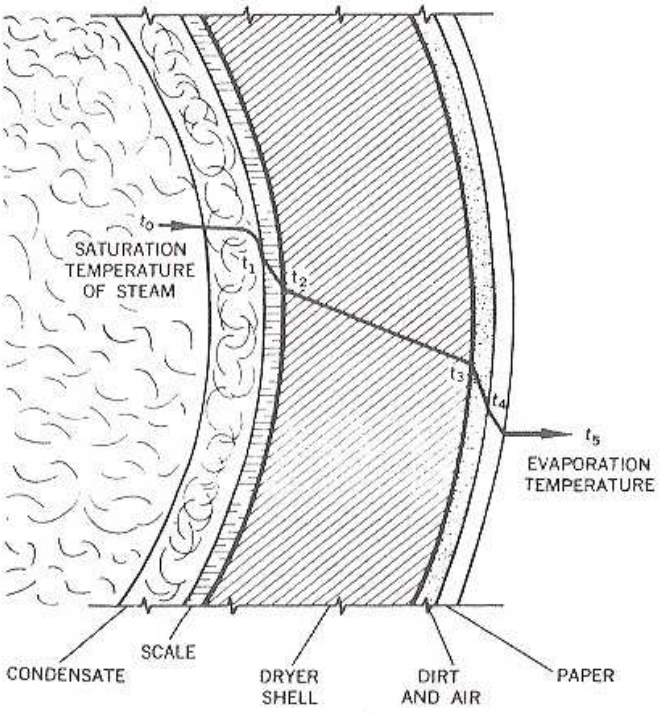


Illustration 2: Temperature profile through dryer cylinder (effect of thermal resistance)

While a condensate layer in slow machines can have a positive effect on heat transfer rates; it can become a real issue for the high speeds of modern paper machines, where condensate layers are immobilized, creating a significant impediment to heat flow. Various techniques have been designed to overcome such problems, like siphon assembly to remove condensate from the cylinder or dryer bars attached axially to the interior surface of the dryer cylinder in order to create peripheral motion in the condensate layer and decrease the thermal resistance.

In addition to local improvements to the cylinders, global solutions have been proposed such as hood ventilation, totally enclosed to provide a much better control of supply and exhaust air flows. The exhaust air offers then a possibility to recover the heat energy supplied for paper drying.

On a general point of view, the industry of paper can't expect a dramatic improvement concerning the drying section performances, unless a different approach to drying is adopted. Some new drying technologies are promising with this regard, namely Press Drying (or hot pressing) and Impulse Drying.

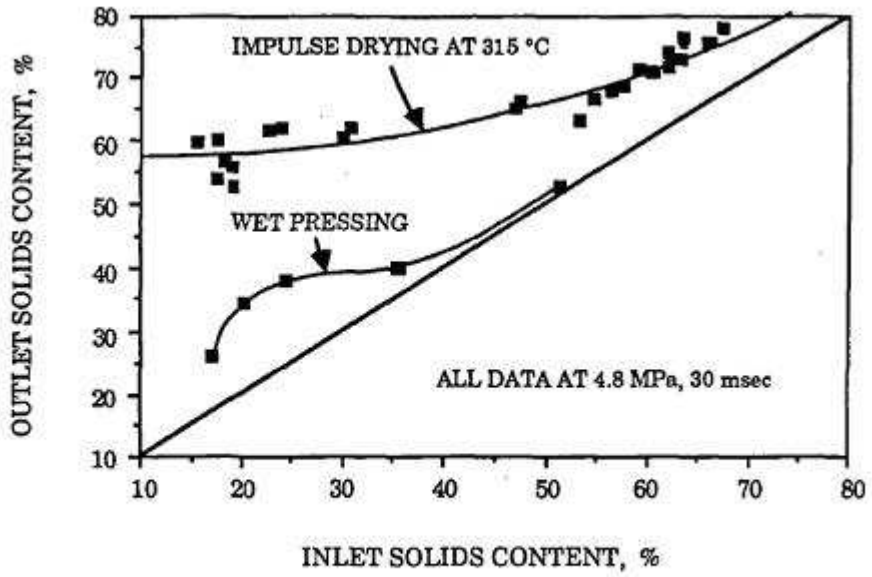


Illustration 3: Comparison of dewatering capabilities - Wet pressing against impulse drying (125g/m² linerboard)

(*In the following, please refer to reference [2] for more information*)

Overview of Press and Impulse Drying

There is a general confusion between hot pressing and impulse drying. However, these two technologies, as close as they might seem, have a completely different range of parameters and effects on paper web.

(*In the following, please refer to reference [3] for more information*)

The term press drying refers to any process that combines both application of pressing and drying, under restraint. This seems to apply to impulse drying as well, however there is a main difference dealing with the range temperatures and pressures applied to the web.

Press drying reaches temperatures and pressures sufficient to reach a temperature above 100°C in the paper web, in order to promote lignin and hemicellulose flow and drying under restraint. The sheets are dried to near the equilibrium moisture content, and as a consequence, drying times are typically several seconds.

Comparatively, impulse drying is a high intensity process that reaches temperatures ranging between 175 to 400 °C, for pressure range in between 4 to 5 MPa. For this process, the residence nip time is generally between 15 to 100 ms.

Press Drying Process

The concept originated in 1925 for the Masonite process and was first considered for use in paper dewatering in the early 70's. For this particular process, the heat transfer rate is limited by conduction, and it's been noticed that the vapor flows toward the hot surface rather than away. The general principle is to make use of belt press with a steam heated cylinder, with exposures time of several seconds.

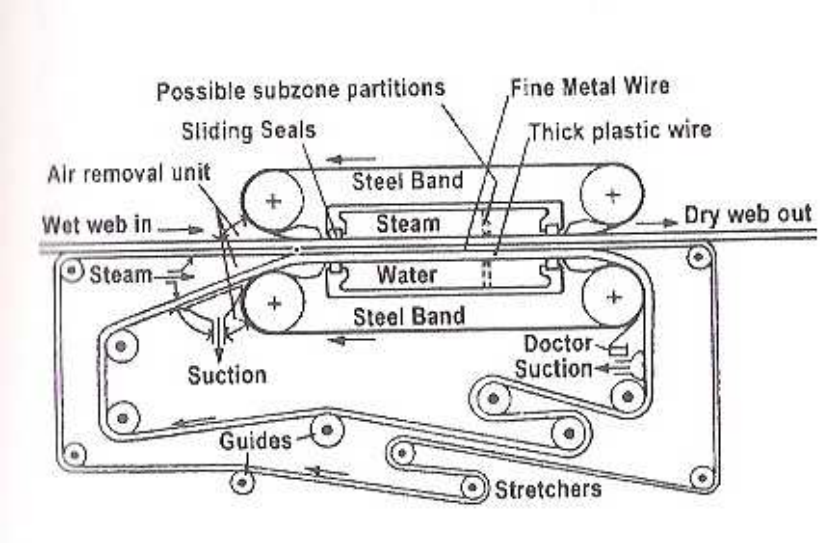


Illustration 4: Illustration of hot pressing - Condebelt process

A quasi-equilibrium vapor generation rate is established by a balance between the heat addition rate and vapor pressure rise due to the vapor flow. Sheet internal temperatures are limited by the vapor pressure rise controlled by the flow resistance from the screens. Overall, the process involves intense vapor phase dewatering. It's believed that there is little opportunity for vapor displacement dewatering or wet pressing. However, bulk vapor flow under a total pressure gradient is likely to occur.

It is estimated that the dewatering rates are to be ten times faster than those of conventional drying. However, only few data exist and these estimations cannot be conclusive so far. A main drawback would concern the total energy consumption which might be greater than conventional drying.

However, among advantages, it is to be noted that elevated sheet temperature, full sheet restraint and long exposure time promote fiber softening and conformability, along with lignin and hemicellulose flow. These conditions lead to excellent densification.

Impulse Drying

A clear way to define impulse drying has been given by Macklem and Pulkowski in 1988 (Reference 1: [25]) Their hypothesis was that impulse drying can be seen as hot pressing followed by flashing of superheated water. This hypothesis has been confirmed in 1999 by Larsson with a series of experiments (Reference 2: [26]).

On a technical point of view, impulse drying is making use of a long press nip with one hot roll to remove water from a wet paper web. The web is exposed to z-pressures up to 4-5 MPa, and hot surfaces are ranging from 175 to 400°C (even 500°C), supplying heat to one side of the sheet.

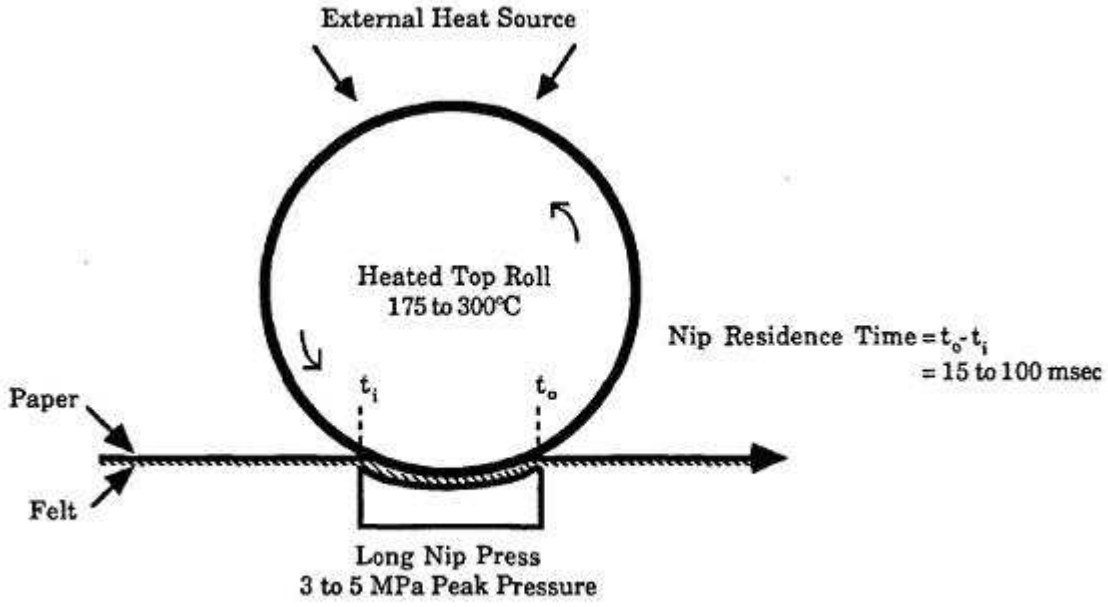


Illustration 5: Implementation of impulse drying

This physics behind impulse drying is now well understood, thanks to major efforts toward that technology since the 80's. This is a two-phase flow process: next to the hot surface, a vapor-filled zone is generated, along with a liquid-filled zone next to the felt.

Liquid is displaced into the felt as the vapor zone grows, inducing a rapid dewatering. The pipe-like heat transfer causes rapid heating through the vapor zone, advantageously producing thermal softening, thus a flow of lignin and hemicellulose. At nip opening, water held by the fibers leaves by a flash evaporation process. The fiber network is extremely well bonded and dense, which means excellent surface properties. Impulse drying promotes surface smoothness, decreased air permeability, greater ink and water holdout and greater surface strength.

Four time intervals have been defined by Wahlstrom, Nilsson and Larsson to characterize impulse drying mechanisms. In the first interval, dewatering and densification are controlled by wet pressing, which means volume reduction. In the second interval, the sheet is filled with liquid, except for a growing layer of pressurized vapor next to the hot surface. As this layer grows in size and pressure, it displaces liquid from the sheet and prevents evaporation of bound water in the vapor zone. Most of the heat energy is delivered by a nucleate pool boiling heat transfer mechanism. During the third interval, external pressure drops below the vapor pressure in the sheet. This allows flash evaporation of water from the fibers. Phenomenon like rapid drying, densification, continued vapor displacement and heat transfer to the lower part of the sheet occur. In this time interval, heat transfer is limited to conduction and vapor convection. In the final interval time, external pressure is dropped to zero, allowing continuing but unrestrained flash evaporation. Internal pressurization may cause bulking of the center zone of the sheet.

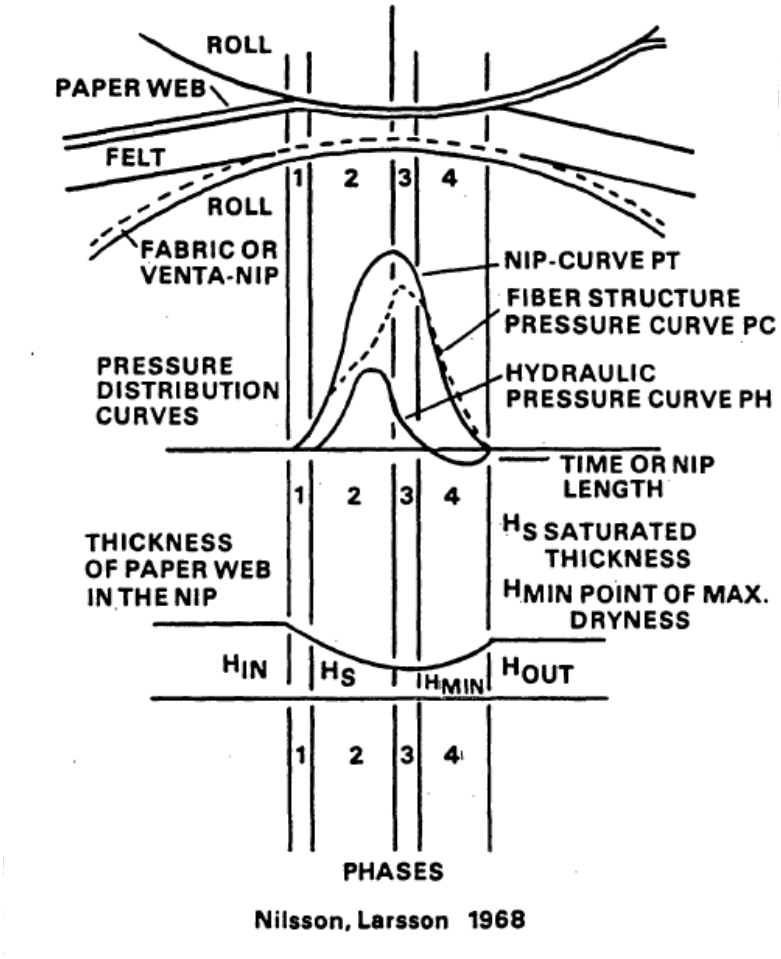


Illustration 6: Pressure distribution and sheet thickness during the 4 phase

The specific energy consumption is defined as kJ per kg of water removed. With impulse drying, energy is used for sensible heating of the wet sheet, then for heating the dry fiber and for evaporation of the water. Clyde H. Sprague suggested in 1987 that values of 550-1400 kJ/kg were likely for typical applications, as compared to 3500-4200 kJ/kg for cylinder drying.

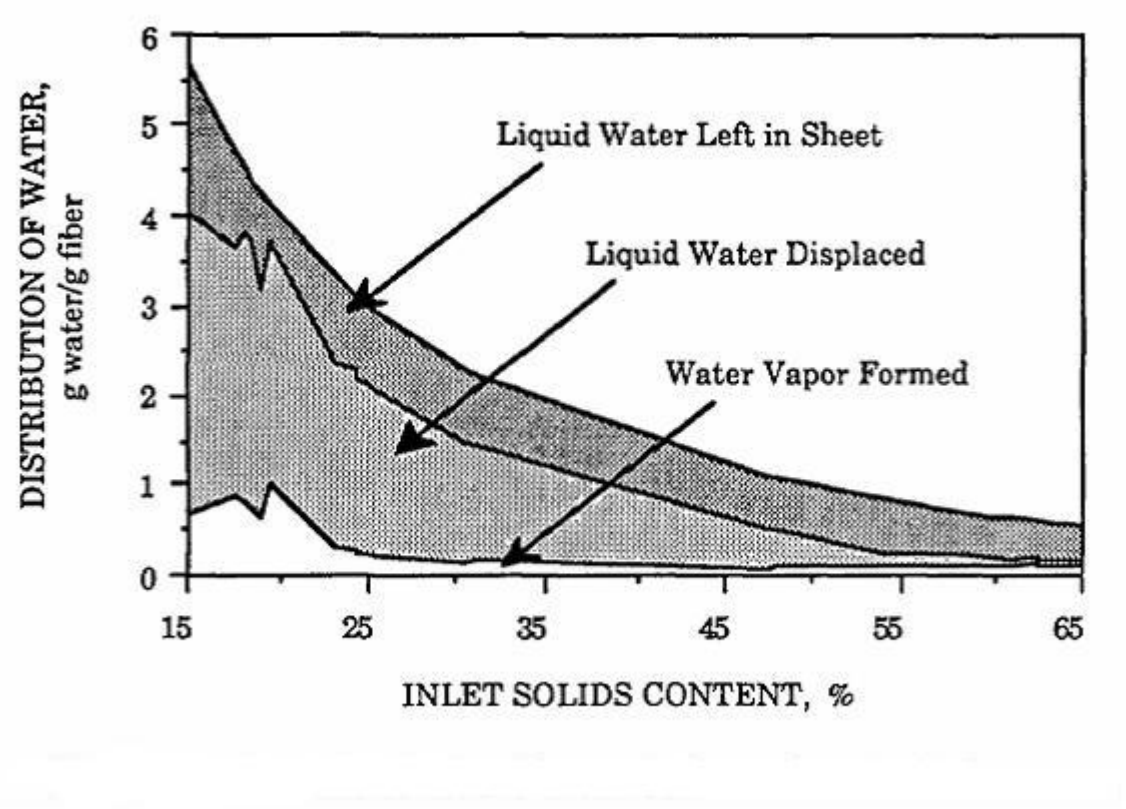


Illustration 7: Redistribution of water during impulse drying (Ref. [4])

Performance of Impulse Drying

Process performance

(*In the following, please refer to reference [4] for more information*)

A summary based on the comparison between these two technologies is important to get a hold on performance in impulse drying, as well as understanding the key parameters, which are to be studied further on in this thesis.

Impulse drying produces process rates which are high enough to allow very short exposure times. In contrast, press drying uses modest pressures and temperatures yielding low process rates which require longer exposure times. This factor is quite an issue when

trying to maintain high pressures for a long time interval while working at full commercial speeds.

Both processes use wet pressing and vapor displacement, but these extend too much greater rate in impulse drying. Flash evaporation of bound water under constraint is an essential parameter in impulse drying for both dewatering and densification. It is not believed to occur in press drying, or only to a modest extent.

Press drying produces a relatively uniform out-of-plane density profile while impulse drying produces a somewhat non-uniform profile that can however be controllable. Resulting density gradients play a critical role in sheet properties such as tear strength and opacity.

A major performance in impulse drying concerns sheet density. The vapor displacement during this process induces dewatering without densification. Added to the fiber collapse during flash evaporation is likely to be useful to produce sheets with dense and bulky center for bending stiffness and z-direction compressibility. In other words, properly controlled, vapor displacement may produce not only very high dewatering rates and dryness, but also a bulky sheet structure. This point is the main concern of this thesis, which will try to approach in terms of methodology and characterization.

(*In the following, please refer to reference [5] for more information*)

As described earlier, flash evaporation is the main feature in impulse drying process. This key element turns out to be an issue in some situations. As a matter of fact, impulse drying commercialization is greatly complicated by the occurrence of sheet delamination. As the nip depressurizes, during the fourth and last time interval of the process,

superheated water remaining in the sheet flashes to vapor and escapes through the sheet surface.

When excessive amounts of energy are transferred to the sheet; drag forces resulting from the escaping vapor can be high enough to overcome the cohesive forces holding the sheet together and the sheet delaminates.

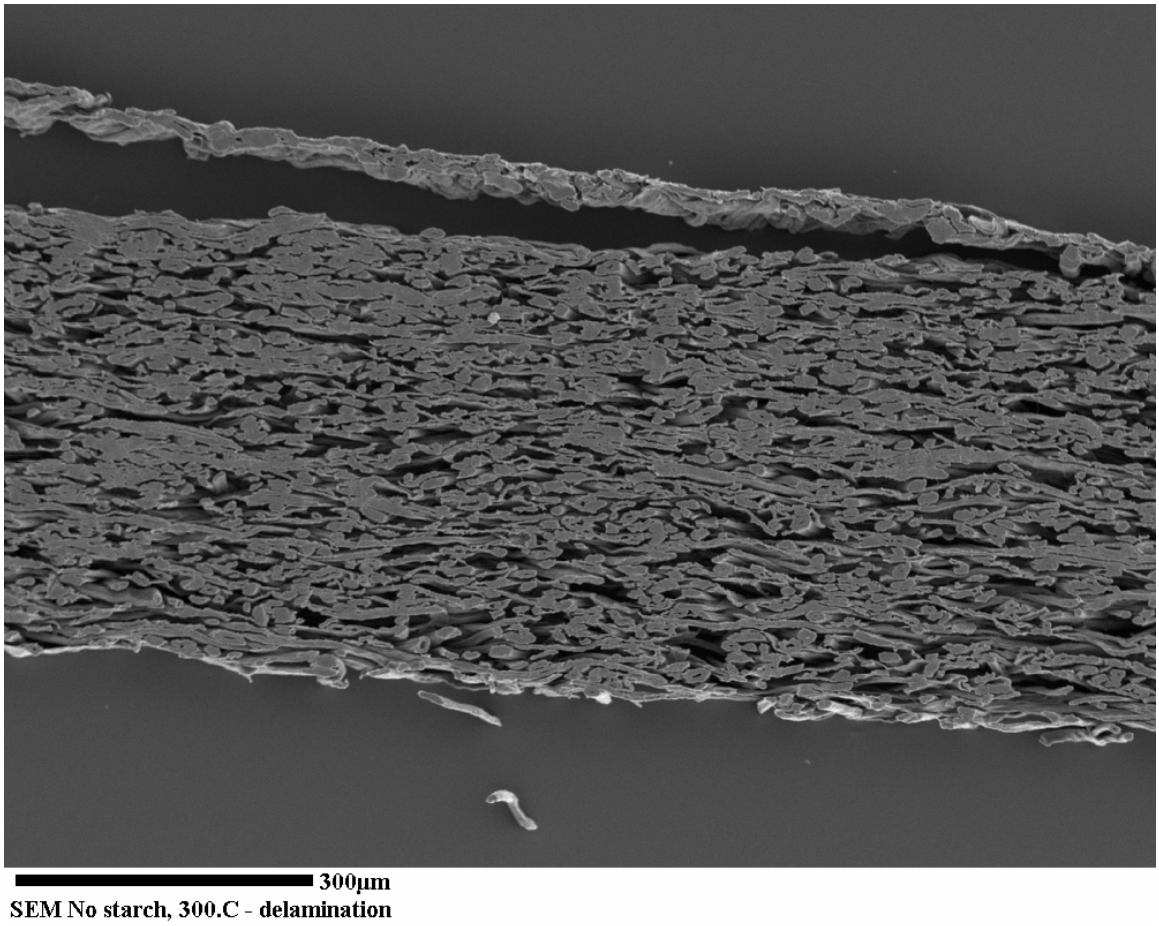


Illustration 8: Delamination captured using S.E.M.

On Sheet Properties and Delamination

In 1992, Dr. Orloff worked on this issue and came up with a solution concerning the heated roll that will be overviewed later on. He suggested that with this solution most of the transferred energy is used to form steam which displaces liquid water, while excessive steam formation leading to delamination could be avoided.

The idea was that delamination could be avoided by controlling the heat transfer to the wet sheet. A set of experiments have been performed both on a laboratory scale and on a pilot-scale.

Water removal is dependent on initial temperature and impulse (or peak pressure), but is independent of the platen thermal properties. Concurrently, it has been found that sheet delamination occurs above a given temperature, that Dr. Orloff has labeled critical impulse drying temperature.

This critical temperature is itself influenced by platen thermal properties and impulse. It is important then to dissociate somehow the study of water removal from the sheet delamination. Both are influenced by the energy transferred to the sheet during the process, but each is influenced by different parameters. As a matter of fact, energy transfer is dependent on thermal mass properties of the platen, which is expressed in [W. $s^{1/2}/m^2$. °C]. In other words, it has been found that energy transfer depends on impulse if the platen material has the properties of "high thermal" mass, while it's independent of peak pressure when "low thermal" mass material are used. This is an important finding since the right choice on the press roll cover material would allow the use of higher peak pressure, thus controlling energy transfer and avoiding delamination, while enhancing water removal performance.

Dr. Orloff has hypothesized that heat transfer is decoupled from wet pressing effects when low thermal mass materials are used, thus avoiding sheet delamination. This not only allows higher pressures operations, but also higher temperatures.

In terms of web consolidation, this means that higher sheet densities can be reached, resulting in higher strength (Specific elastic modulus, compression strength, burst strength …)

Further experiments were then performed to characterize apparent sheet density in terms of relative strength; especially bond strength characterized by the specific elastic modulus. It's been concluded that both parameters appear to be independent of platen material. A similar linear relationship was preserved between these two parameters, regardless of the material used. It's been also noted that as long as temperatures are below the critical temperature (thus avoiding delamination) no internal bulking occurs.

This remark truly defines the purpose of this thesis as a continuity to these experiments and conclusion. The goal is to investigate the potential of starch in paper web when impulse drying is used, under the hypothesis that bond strength will either be kept or reinforced, thus avoiding delamination ,while operating above critical temperatures to trigger internal bulking.
This thesis intends to approach this second key parameter with a new point of view, again on a methodological stem point. The idea is to come up with key parameters from consolidated paper webs to give a new opportunity, dealing with impulse drying.

Critical Parameters on Heat Transfer

(*In the following, please refer to reference [6] for more information*)

It has been hypothesized that during impulse drying, water in the web is heated and pressurized such that it exists as a subcooled liquid just prior to nip opening. As the nip is opened, the water in the web continues to be heated by contact with the hot press roll surface while experiencing a sudden drop in restraining pressure. These conditions lead to a sudden increase in internal web pressure and rapid flashing of the subcooled liquid to vapor. The pressure difference between the inside and the outside of the web can cause the disruption of the web. In addition, vapor that lacks a clear path to exit the sheet and is trapped inside the web prolong the time during which this extreme pressure difference is

experienced. The net result is that the fiber network ruptures, resulting in the phenomenon known as delamination.

This statement by Dr. Orloff truly guides the subject here. From a series of experiments, insights are to be determined in order to fully address the potential of a technology. Impulse drying has been studied with increasing precision, leading to the creation of a pilot-scale model that offers the possibility to analyze the drying process on an 'industrial' scale. As stated previously as well, impulse drying is a high intensity process, therefore putting the paper web under extreme conditions. Dr. Orloff's statement defines the logical approach to such situation. Now that the technology is fairly understood, it is important to get specific details on the physics behind drying during impulse drying process. The most logical approach is to study the heat transfer process, in terms of heat flux as well as phenomenological impacts. It is only natural to formalize a model from the observations taken from experiments. This will serve the purpose of understanding as well as feedback to further studies. The following part of this thesis is pursued in this logic: from previous series of experiments concerning impulse drying and drying processes in general, hypothesis are formalized for the purpose of modeling, which will in turn be formalized in numerical studies in order to bring a more precise understanding to the subject, and back to more experiments to draw general conclusions on assessing a variety of issues concerning the process of drying.

Boiling Heat Transfer and Pore Size Distribution

(*In the following, please refer to reference [2] for more information*)

Impulse drying employs phase change heat transfer to dry a paper web. The fundamental mechanism during this process has been defined as boiling heat transfer. At surface temperatures that exceed 25°C, heat flux is controlled by the rate at which the fibrous bed

supplies water to the heater surface by capillary forces. It is therefore directly related to the pore size of the paper web. Interestingly, it's been found that the heat flux increases significantly at pressure levels lower than what is likely to exist within the sheet during impulse drying.

Pore size distribution in the fiber web is believed to be as critical a parameter as internal pressure during impulse drying. High heat fluxes sustained during the process are likely to be related to this distribution in the fact that pore distribution controls the flow rate of water to the plane of evaporation within the sheet. Pressure helps maximizing the water supply mainly by keeping a highly saturated moisture level within the sheet, by increasing vapor density.

This paper constitutes above all a study of the impact of starch in the paper web, under high pressures and high temperatures conditions. It underlines also the challenges inside our principal material of study: the paper web. Even though paper structure hasn't been the first subject of study, essentially because of the difficult task it represents; some suggestions were underlying its importance to the process performance. The above observations have leaded to an extensive study on the so-called boiling heat transfer in porous media.

Understanding Heat Flux during Impulse Drying

(*In the following, please refer to reference [7] for more information*)

As suggested above, heat transfer during impulse drying has first been approached with the study of boiling heat transfer. Various observations have pointed in the late 80's that the so-called pool boiling heat transfer was prevailing over the more common convective boiling process. The fact that impulse drying is used over a porous medium points out

also the importance of such structures, and it has been observed by Gary Rudemiller in 1989 that parameters such as pore diameter and system pressure were of great importance on the performance of drying.

An extensive study of pool boiling heat transfer has been performed in order to formalize the future of study concerning impulse drying. Hence, Gary Rudemiller has pointed out the fact that there exist two main regimes in porous media. The boiling heat transfer is first initiated by the nucleate regime; where heat flux is directly related to the so-called wall superheat. The wall superheat is defined as the difference between the heater surface temperature and the saturation temperature of the liquid. Therefore, nucleation is controlled by the heater surface, while physical limitations for vapor expansion are posed by the rigid porous structure. This has a crucial importance with respect to impulse drying since it can operates to high level of temperatures, hence its capacity to trigger several nucleation sites. In other words, there is no influence of internal pressure at that stage of impulse drying. After a transitional regime, an iso-heat-flux regime take over the first one, where heat flux is almost constant and no more dependence on wall superheat are observed. At this stage, the magnitude of heat flux is controlled by the rate of liquid supply to the heated surface. Hence here the observation on pore diameter influence as well as internal pressure.

Rudemiller pointed out also the importance of the vapor film formation and the phase change during the iso-heat-flux regime. Numerous invaluable information are present in this study, and are truly pointing out the variety of approaches one can take in order to study impulse drying and its performance on porous structure. As a general approach, Rudemiller has described the potential of heat surface characteristics that have been thoroughly studied later on by Dr. Orloff, as well as the importance of multi-phase flow in porous medium. Hence, his idea was to increase the number of nucleation sites in order to take advantage of high performance boiling.

The essential parameter is the impact of applying heat from above the paper web. It has been observed by Udell that during boiling heat transfer, the porous bed is segregated into three zones:

The top zone is filled with vapor, and heat transfer is dominated by conduction

The transition, two-phase zone is a counterpercolating nearly isothermal zone. Processes of evaporation, convection and condensation occur simultaneously in this particular area.

The third zone is a water saturated zone.

It is finally observed that generated vapor convects downward due to pressure gradient (and probably buoyant forces) while liquid flows upward under capillary forces influence, acting thus as liquid resupply to the heater surface. This last observation is of crucial importance since it points out the importance of venting of vapor which can be either a pushing front to dewatering or a thermal limitating factor to the heater surface, depending on the performance of venting.

These parameters were at that time still to be investigated, but boiling was believed to be active throughout the entire event of impulse drying, at various rates, sustained by counterpercolation of steam and liquid.

CHAPTER 3

EXPERIMENTS

A first series of experiments have been performed by Isaak Rudman in order to investigate the issues of delamination during impulse drying. After briefly studying the capability of a wet paper to resist “peeling”, the idea was to somehow reinforce the inner structure of handsheets. For this purpose, a study of different starches has been done, since starch is a material widely used in the paper industry as a sizing and strengthening agent. Various experiments are chosen to characterize the advantages in terms of strength and bulking, along with specific impulse drying conditions.

Preliminary Tests and Equipment

Foreword on Experimental Conditions

The choices of parameters for the experiments are defined as suggested in the first part by the set of experiments run by Dr. Orloff in 1992 on the pilot-scale testing concerning delamination.

However, the handsheet prepared in the lab were not preheated as they had been for the pilot-scale experiments (the preheating temperature was 90 to 100°C for the latter).

For the impulse drying, the felts were conditioned to a moisture ratio of 15%, the dwell time was fixed to 40 ms. and the ingoing temperature range of the hot platen was between 150°C and 300°C. The peak pressure was maintained as much as possible to around 5.4 MPa (about 5378 kPa), accordingly to a previous set of experiments performed in 2005. For these experiments, Isaak Rudman focused on the study of different starches, along with different nip residence time and platen temperatures as described in the following table.

Process Conditions (2005):

Table 1: Process conditions for impulse drying on starch imbued handsheets

	Nip Residence Time [ms]	% Ingoing solids	Temperature °C
cases	30	35	298
	30	35	25
	110	35	300
	110	35	25
	30	35	25
	30	35	250
	30	35	310

163 samples were prepared and impulse dried for this study, which allows a fairly good basis for averaging the results.

Concerning the preparation of handsheets, following the trend of previous experiments, the same furnish was used, which was southern hardwood Kraft from Alabama River Pulp Company, bleached fibers. This furnish will be the same for all the future experiments, and all samples prepared with or without starch

The desired grade targeted was linerboard, hence a basis weight of 250 g/m² chosen for the entire set of experiments. The sheets were prepared using a square mold at the I.P.S.T. following the designated TAPPI procedures (dimension 8.1" by 8.1"). They were then pre-pressed to reach ingoing solids in around 30% - 40% for the impulse drying and wet pressing experiments (pressing at room temperature – 25°C).

The square handsheets were then cut to circular samples of 4" diameter.

Continuing in the same trend of experiments from Dr. Orloff, as well as those performed by Isaak Rudman, a level of refining at 600 ml. (CSF standard) has been maintained for the preparation of handsheets.

(*In the following, please refer to reference [5] for more information*)

The work was performed using a laboratory-scale electrohydraulic press to simulate impulse drying. A ceramic-coated platen with a thin, fast, vacuum deposited copper/nickel thermocouple was mounted in the press. This equipment (labeled M.T.S.) has been studied and proved to simulate the conditions of impulse drying on a laboratory scale fairly well.

The goal of this thesis is to investigate the potential of starch imbued handsheets in terms of strength (in order to avoid delamination) and bulking (trying to somehow reach a "puffing" effect, avoiding too much densification). As a fundamental parameter to delamination has been determined to be the so-called critical impulse temperature, the first approach has been to fix the impulse (area under pressure-time curve) and study the effect of temperature on the handsheets. For this purpose, a set of temperature has been studied (besides wet pressing at 25°C – Room temperature): 150°C, 200°C, 250°C, and 300°C. The idea behind was to trigger expansion of starch inside the handsheets, thus enhancing the bulk properties.

Handsheets Preparation

As said before, the furnish was the same for all sets of experiments, the question of refining did arise, and a brief study on different refining level has been performed, without being conclusive. Defiberization has been the only process kept for this reason, to reach a level of freeness of 650 ml. CSF.

As said previously, handsheets were prepared in the lab using a square mold using the TAPPI procedure T205, and under three different cases. For the first case, labeled the control case, the preparation of handsheets was without starch. The second case labeled the 10% case is for handsheets containing 10% starch (calculated on a basis weight). The last case is the 20% case, which corresponds to an amount of starch of about 20% by total weight. A particular attention has been brought to the introduction of starch in the slurry. It turned out the best way to mix starch with the slurry was to cook it before preparing the handsheets.

The square handsheets prepared were 8.1” by 8.1”. They were pre-pressed to reach 30 to 40% ingoing solids, using a pressure of 425 kPa for 10 seconds. Once pre-pressed, the sheets were cut into four circular samples of 4” diameter and properly labeled for data recording. They were all bagged individually in order to store them before impulse drying experiments.

Preparation of Starch CP 3005

This step is a key step in our series of experiments since starch is the main parameter in studying the effect on fiber bond out-of-plane strength in parallel with bulking effect. Under previous experiments (from Isaak Rudman), starch was prepared at 8% consistency during cooking.

A choice of one liter of cooked starch was made. That was 80g of starch powder to add. The powder was estimated at 90% solids, so 10% of powder was added to the previous calculated amount, i.e. 88g total.

The mixture starch powder and water was cooked up to 78 – 80°C and then kept at that temperature during 20 min. Problem of evaporation needed to be accounted for.

A rigorous control of cooked starch consistency was thus important for the further preparation. It has been found that cooked starch can be stocked and re-used later if kept

diluted down to 2 – 3% consistency. A rigorous control on these diluted samples consistency is important for further use in handsheet preparation (our cases of study includes 10% and 20% of starch with respect to the total weight of sample prepared at 250g/m²).

The diluted cooked starch (with deionized water) was then poured in the mold with the right amount of slurry, before draining the mold.

Summary of Previous Experiments

2005: Splitting Device – Test on delamination at controlled temperature and relative humidity.

Isaak Rudman was in charge of a series of experiments intended to analyze wet sheets split with a specially designed splitting device. The wet sheet splitting device was designed to measure out-of-plane cohesion force of wet sheets.

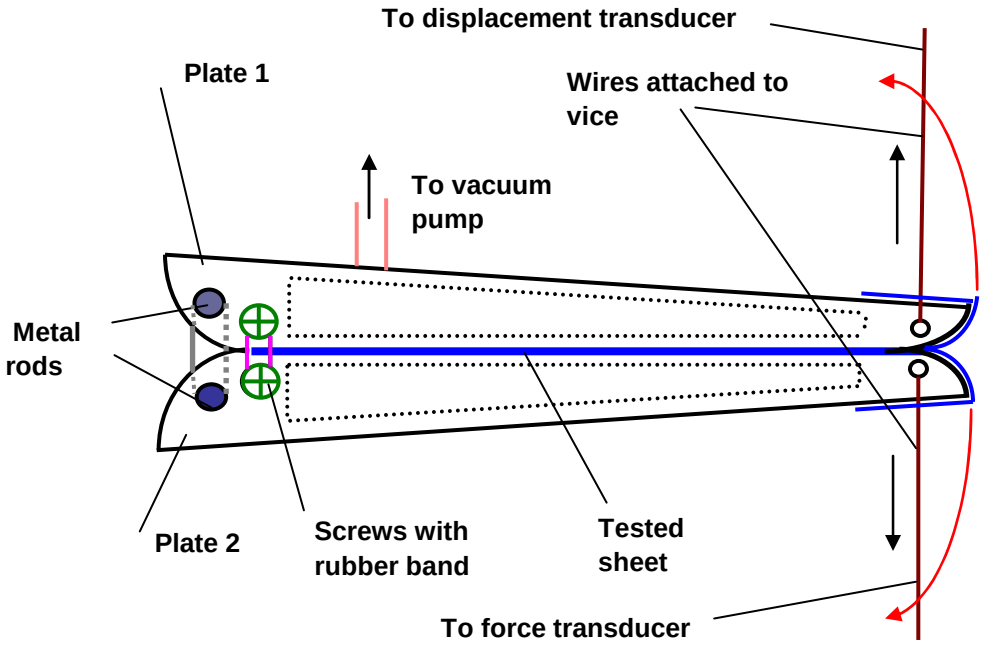


Illustration 9: Sheet holder splitter (Top view)

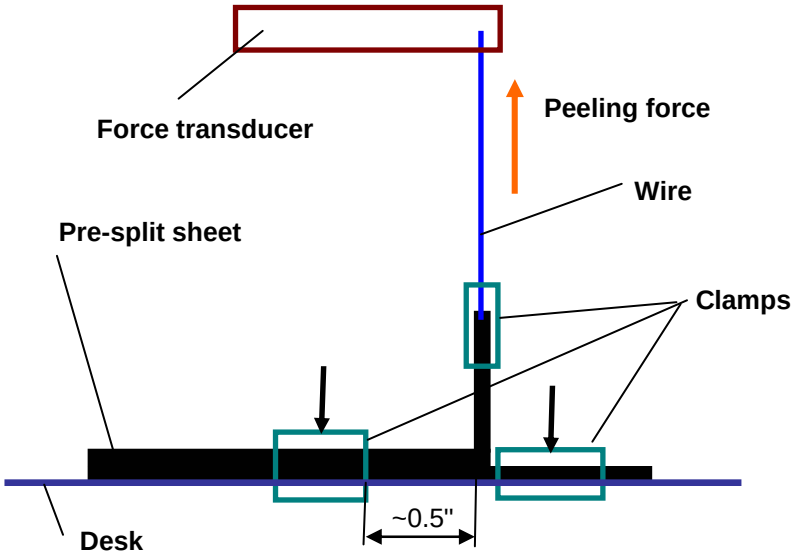


Illustration 10: Schematic of peeling device

Brief results were obtained to further our understanding of bulking and enhancing fiber bond strength.

As detailed previously, a somewhat surprising idea was to use starch in a significant amount along with the preparation of the handsheets. This has lead to the study of different type of starches in order to determine the best choice.

Determination of optimal starch in specified conditions of use (cooked, uncooked, modified, unmodified) was provided later on with the series of ultrasonic testing in February 2006. The significant amount of samples prepared by Isaak Rudman (163 samples) has been of fair advantage to conclude on repeatability and averaged results per case.

From Isaak Rudman: Report on Bulking, Dec. 12th, 2005

Modified corn starch CP5572

Cationic corn starch CP5960

Unmodified pearl corn starch CP3005

Unmodified wheat starch, which has smaller particle size and can be possibly absorbed in uncooked state.

Each of these starches was used in uncooked and cooked condition. Uncooked starch solution was obtained by diluting dry starch powder in deionized water at temperature of about 40-50 ^{o}C. The concentration of starch solution was about 8%. The starch powder was presumed to have moisture content of 10%. Corn starches were cooked at maximum temperature of 78 ^{o}C in double-cooker while mixing manually. When no increase of viscosity was observe the cooking was over. Once the temperature reached 78 ^{o}C, the cooking lasted for 5-8 min. Pearl unmodified corn starch CP3005 had the highest viscosity after cooking. Unmodified wheat starch showed lower increase in viscosity and was cooked at temperature of 83 ^{o}C. As some moisture evaporated during the cooking, the concentration of starch increased from initial 8% to about 10%.

[…]

Adding starch into the slurry yields the sheet with higher starch content. The cooked corn starch CP3005 and cooked wheat starch may be proper choice. They have a potential to produce a foamy structure of different pore size (smaller for wheat starch because of smaller molecule). In this case, a number of issues may arise. First, retention of the starch in forming section may be different than in handsheet mold. Second, sticking in pressing and beginning of the dryer section may be a serious issue.

The change of the sheet bulk dB can be determined as a ratio of the difference of sheet thickness to weight of removed water:

Equation 1: Change in sheet bulk

$$dB = (t_{in} - t_{out})\, A / dm$$

Obviously, lower ratio would indicate that lower decrease of sheet bulk occurs. Thus, by comparing dB for various sheet and pressing combinations some conclusions as to how the bulk changed can be derived. Apparently, changes in the bulk should affect the parameters of the porous structure (pore size, interconnectedness, etc.).

Increase of impulse dried sheet bulk is to occur if intensive flash evaporation happens. When vapor is formed, it is driven by pressure differential and escapes the sheet at levels which are above saturation temperature and condenses in portions of the sheet which are below saturation temperature defined by internal pressure. Obviously, in portions of the sheet where intensive evaporation occurs, sharp decline of sheet temperature is to be expected. And opposite, intensive condensation of the vapor, would lead to significant rise of temperature in a given portion of the sheet. Thus, intensive flash evaporation is accompanied by characteristic spikes in temperature curves.

*The formation of foamy samples in heated to 230 ^{o}C mold under pressure of 508 psi (3.5 MPa) and pressure time of 10 s, described in Glenn and Orts (2001) (*Ref.[11]*), indicated that the mixture of uncooked and cooked starch was used when producing highly porous foams. The density ranged from 0.97 g/cc at ingoing solids of 89% to 0.078 g/cc at solids of 84% and lower up to 80% of solids. At lower solids, formation was poor and accompanied by excessive expansion and sticking. Increase of bulk with increased moisture content may be evidence that more intensive evaporation took place when pressure was rapidly released which facilitated to forming foamy structure.*

As we can see, the original working hypothesis was that a foamy porous structure was developing in the starch imbued handsheets under impulse drying, due to the capacity of starch to expand under high temperature and high pressure.

Ultrasonic Testing

January 2006: Directly following the series of experiments by Isaak Rudman, a series of ultrasonic testing was performed in order to investigate the average specific out-of-plane elastic modulus which characterizes the out-of-plane strength of the dried sheet. Along with this parameter, average caliper was measured as well. It is useful information to provide an estimated value of bulk:

In order to have significant parameters, all the values were thus normalized to the calculated Basis Weight. Therefore, all the samples were carefully conditioned for testing.

Following the TAPPI procedure on conditioning, all the samples were prepared in an environmental controlled room. This lab is divided in two rooms, both controlled at 73°F, but with different relative humidity. The samples were first kept in the pre-conditioning room, which is maintained at 20% relative humidity (dry room), over night. Then they were transferred to the conditioning room, maintained at 50% relative humidity, for 24 hours.

The next step was therefore to calculate each individual sample basis weight for both purpose of normalization and ultrasonic testing (data needed for the testing).

The first series of ultrasonic testing was intended to record the specific elastic modulus averaged over five measurements per sample. This way of measuring data has the advantage to minimize errors such as those occurring because of stress concentration, occurring because of the way handsheets were impulse dried. The following equipment was used to measure out-of-plane stiffness.

Illustration 11: Ultrasonic tester at I.P.S.T.

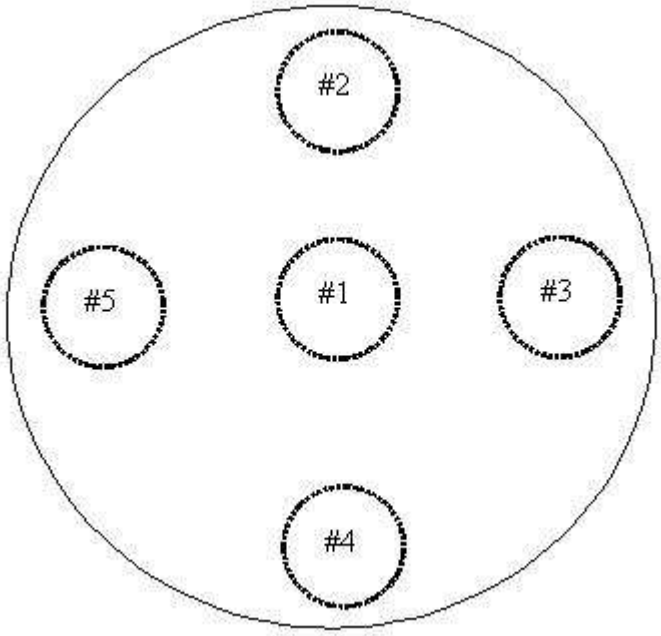


Illustration 12: Position of ultrasonic sensor on circular samples

The handsheets were all tested in the same fashion, and each measurement taken into account for averaging.

Following are the results of testing (completed by a series of retesting for some cases that weren't clear at the first approach). It allowed us to determine the best choice of starch for further experiments while confirming the trend expected in strength and bulking with respect to impulse drying (as opposed to wet pressing).

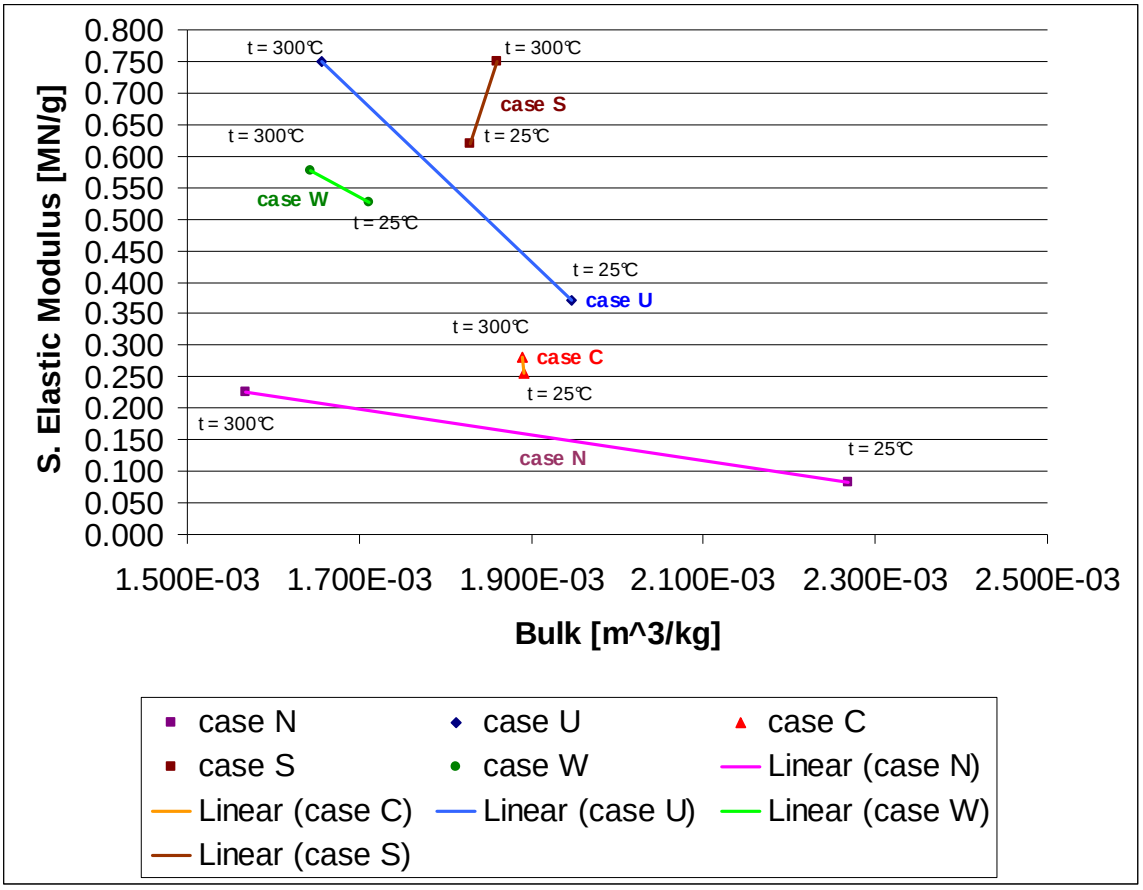


Figure 1: Results of ultrasonic testing on starch imbued handsheets. Specific elastic modulus vs. Bulk

Table 2: Summary on starch used with the optimum choice in blue

CASES:	N	No starch.
	U	Uncooked cationic starch.
	C	Cooked cationic starch.
	S	**Cooked Unmodified Cornstarch.**
	W	Cooked Unmodified wheat starch.

Our goal is to increase the relative bonding strength of the web, while enhancing the bulk of the paper sheet as well. The only satisfactory case for this purpose is the cooked unmodified corn starch. All the other cases (except on the unconvincing results for cooked cationic starch) were exhibiting a lost in bulk while strength was gained. The next step in the study of starch imbued handsheets was then the investigation of the influence of impulse drying temperature.

Influence of Platen Temperature – Results

March 2006: A series of impulse drying testing was conducted to study the influence of impulse drying temperature on both strength and bulking for starch imbued handsheets.

The range of temperatures which was considered was: 25°C (wet pressing, as a control value), 150°C (not conclusive, results not presented), 200°C, 250°C, and 300°C.
The cases of study are the usual: no starch, 10% of starch and 20% of starch based on the desired basis weight ($250 g/m^2$ – The amount indicated are relative amounts retained during handsheets formation).

In parallel, several parameters were recorded in order to assess the effect of impulse drying on handsheets reinforced with starch. Dewatering was one of these parameters, and for this purpose, ingoing and outgoing solids are recorded in lab during impulse drying that I've conducted in march 2006 (See figure 2).

Following are the results for dewatering:

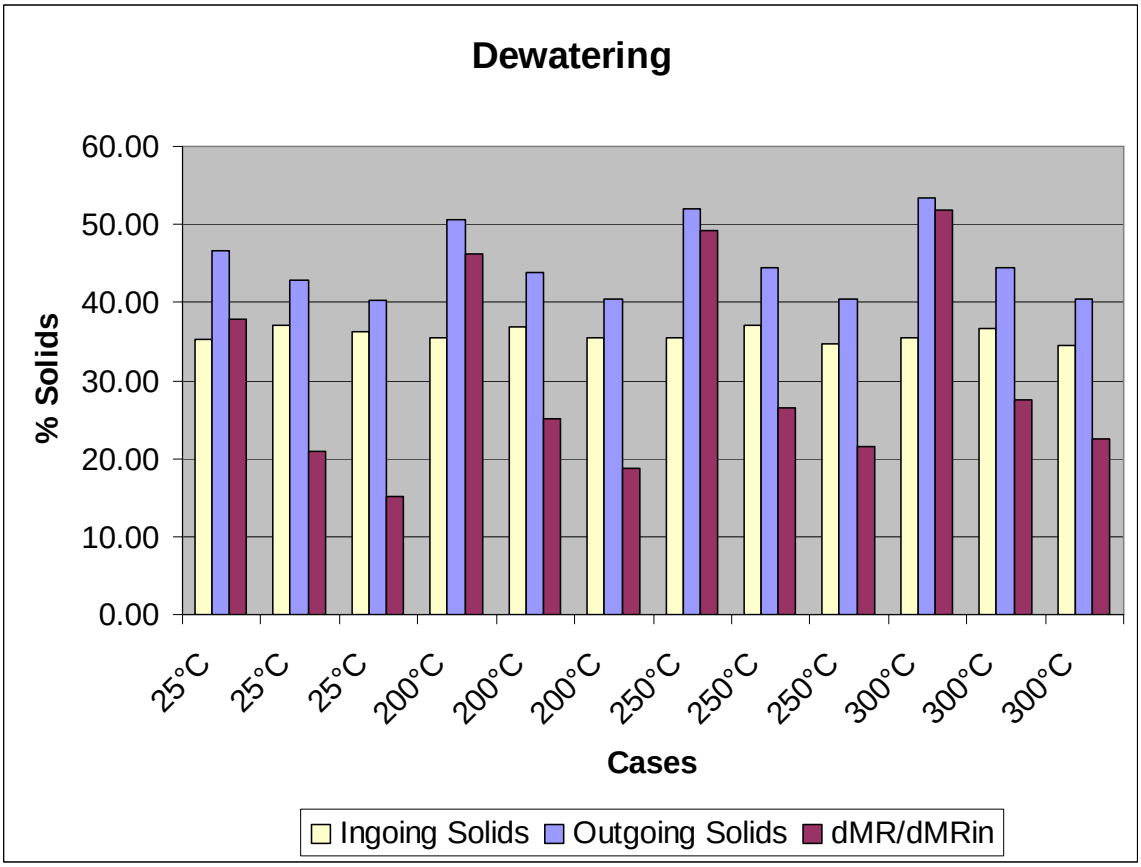


Figure 2: Dewatering. 3 cases of relative starch content per temperature (0%, 10%, and 20%)

Here, the purple data indicates the relative capacity of dewatering for a given case, with respect to the ingoing solids. For each case of temperature, the bars refer to respectively 0%, 10% and 20% of relative amount of starch in the handsheet. There is clearly a decreasing capability of dewatering for each case of temperature as we increase the amount of starch in the handsheet. This is a further element to our hypothesis of closed matrices for the case of starch imbued handsheets. We can also observe the influence of impulse drying temperature on the dewatering capacity. There is clearly a better performance with higher temperature, which correlates to the softening of fibers and starch.

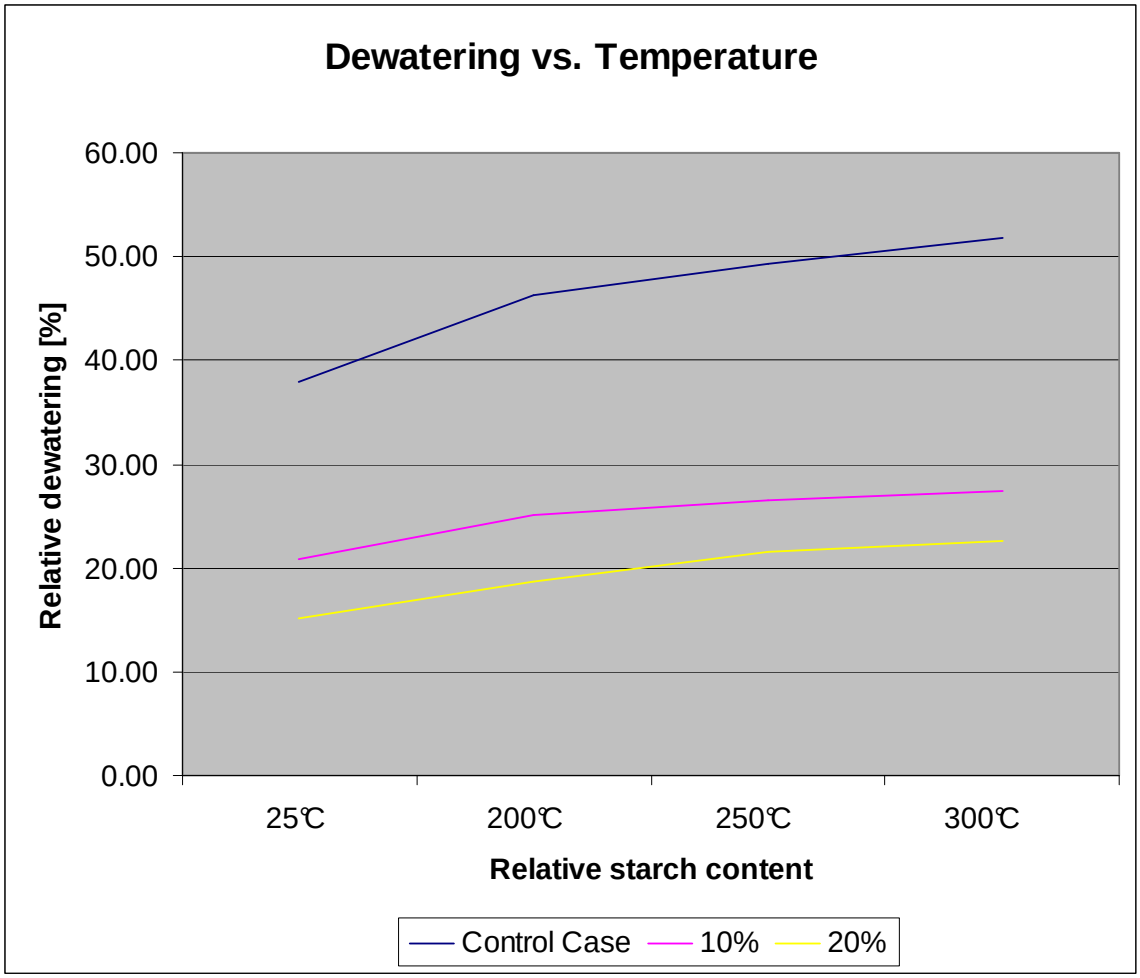


Figure 3: Dewatering with respect to temperature

We observed that dewatering follow the same trend, regardless of the relative amount of starch in the handsheets.

The impact of starch on dewatering was clearly visible here. The dewatering capability of handsheets drops by almost half in each case of temperature between the control case and the starch imbued handsheets. This might be seen as a drawback in terms of impulse drying use, but compared to the trend concerning strength and bulking; a tradeoff has to be considered.

Hence, this dewatering drop can be interpreted as the mechanism behind bulking, as Dr. Orloff has hypothesized it. If we were to relate bulking with starch, besides the fact that this polymer has a tendency to expand under high temperatures conditions; the fact that dewatering is lowered down could be the triggering effect for bulking. Water can indeed be trapped in pores during flash evaporation, inducing then the observed bulking in starch imbued handsheets. If this is the case, by observing the porous matrix of different samples, we should be able to determine if starch imbued handsheets are indeed “more closed” than regular handsheets.

For this purpose, one has to study the general connectivity of the different porous structures involved in this series of experiments. This is where scanning electron micrographs have an important role to play. Using image analysis, one can characterize the connectivity of each structure, along with other key parameters of porous media. If indeed, the connectivity of starch imbued handsheets is significantly lower compared to regular handsheets, then further experiments will be needed to confirm the hypothesis, or bring another explanation if necessary.

Below are the results concerning the study of out-of-plane relative strength as well as bulking effect.

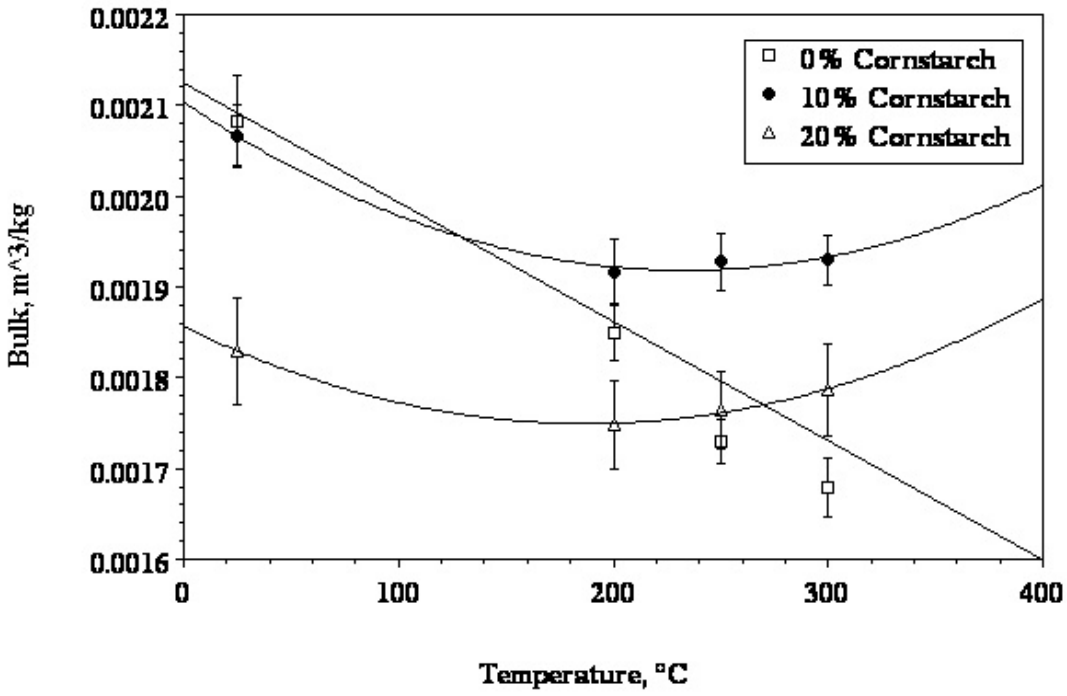


Figure 4: Bulk vs. temperature for the three cases of study

From these results, we were able to infer that there is indeed a potential with starch imbued handsheets compared to regular handsheets when submitted to impulse drying conditions. There is clearly a potential when the temperature of impulse drying is reaching 250°C and above. The best results seem to be reached with only a small content of starch for this particular grade (250g/m²). It would therefore be interesting to try to determine the optimum amount of starch with respect to temperature and grade.

This said it is important to double check the bulking effect with out-of-plane strength. One wouldn't like to gain in bulk while losing in strength.

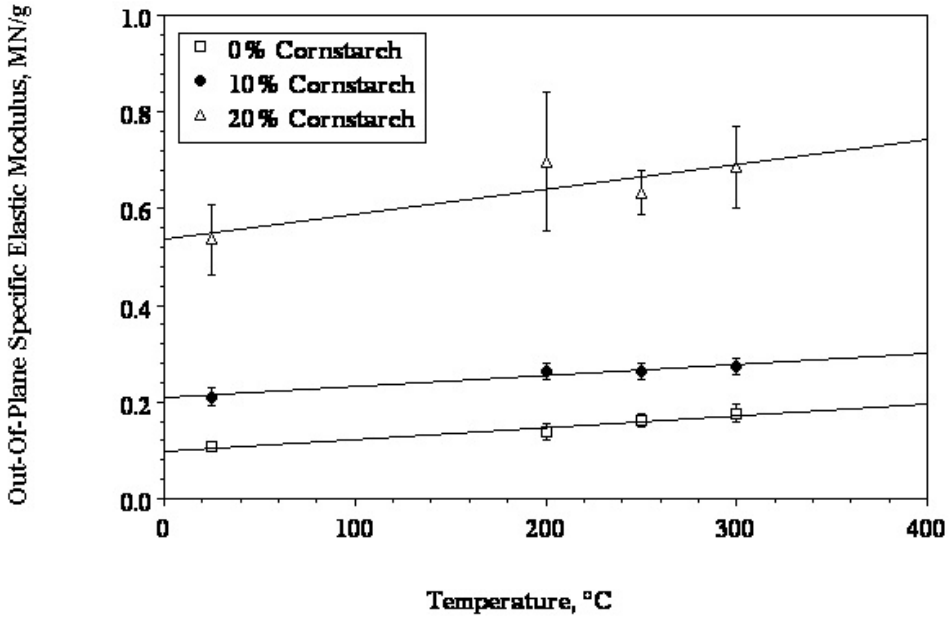


Figure 5: Specific Elastic Modulus vs. temperature

Clearly, analyzing these results, using starch in handsheets for impulse drying can only lead to a benefit in strength. As the trend appears here, it seems that the more starch there is in the handsheet, the better it is. The same conclusion is considered with respect to temperature, the higher it is, and the better it seems to be.

Comparing relative strength to bulk should give us a fair idea on the possibilities in terms of starch content and temperature range.

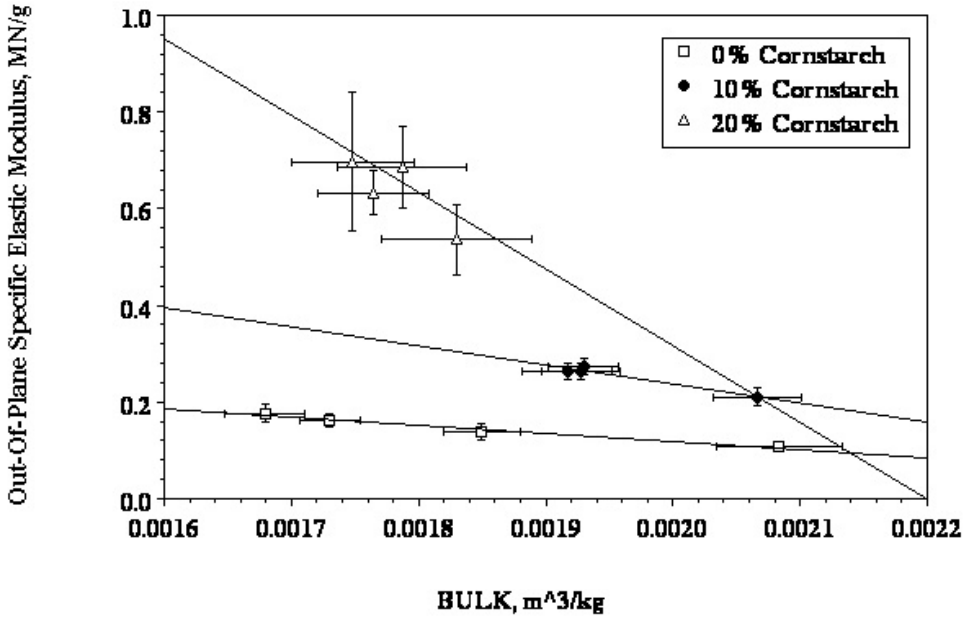


Figure 6: Specific Elastic Modulus vs. Bulk. All experimental temperatures considered

As expected, when considering impulse drying on the control handsheets, bulk is gained only when there is a loss of strength.

Interestingly, wet pressing ends up with a kind of same effect with the addition of starch. In other words, using wet pressing on starch imbued handsheets doesn't lead to the desired expectations: a 10% relative amount of starch handsheet once pressed will have poor strength for a high bulk, although showing almost a double value of strength compared to the control handsheet. A 20% relative amount of starch handsheet will show low bulking for a reasonable strength, but again only in the worst possibility as compared to impulse drying.

Besides these conclusions, one can notice that there is a real tradeoff between the two cases of starch amount while considering impulse drying. With a "low" amount of starch, a high bulk is reached for a reasonable gain in strength, while the "high" content of starch show a good gain in strength for a reasonable gain in bulk.

Brief Analysis on Starches – Further Experiments

In an effort to characterize the impact of high relative starch content usage in handsheets, a series of experiments was run under conditions of wet pressing, with the idea of controlling the homogeneity of starch mixing without the influence of temperature (in other words mixed at room temperature and dried under wet pressing conditions). This intention is to study the worst case of starch integration; without its capability of expansion under high temperature, nor the potential of plasticizing of furnish under such conditions of temperature as well.

Nov. 2006: Bulk and Elastic Modulus – Wet Pressing, control of Starch Integration.

Efforts were expended to reproduce the experiments from the period Nov. 2005 to March 2006.

A different pressure profile was chosen to further investigate differences in bulk and out-of-plane elastic modulus for the three cases of study: control case (no starch), 10% starch and 20% starch imbued handsheets.

A rigorous procedure was used in order to prepare cook starch for handsheet preparation.

A systematic control of solids content was used to minimize errors in the final handsheet basis weight. As opposed to the early series of experiments, no ply tissue was used in an effort to observe the possible sticking of handsheets on the press platen. This wasn't the case in the series of experiments in 2005 in which a problem o sticking occurred during impulse drying, overcome at that time by the use of ply tissue.

Main differences compared to wet pressing case in 2005 (performed by Isaak Rudman):

Corn Starch was prepared under standard procedures; however a closer control was put on consistency determination. It was found that preparing the cooked starch solution at 2% to 3% consistency was a good way to keep starch samples for reuse.
Starch was added to slurry under the assumption that respectively 90% and 80% of previous amount of fibers was kept in the sample of slurry. The correct amount of starch was determined at each trial in order to reduce the errors in the final basis weight.

Samples were pre-pressed to about 30% ingoing solids instead of 35% from the previous set of experiments. Felts have been prepared to 15% of moisture content as done in previous set of experiments.
The previous pressure profile (which shows a typical sharp pressure gradient release at the nip opening, for a peak pressure of 5.4 MPa and a nip residence time of 40 milliseconds) has been tested and validated compared to the previous set of experiments before choosing a different one for our purpose: a lower pressure gradient at the release was chosen, with a final ramp in order to modify out-of-plane sheet response. This was major difference compared to the previous set of experiments since the area under the curve was not the same anymore (hence a different relative energy delivered to the paper web).
A wire was however used to keep consistent with the ultrasonic testing issues encountered in previous set of experiments (essentially zones of higher stress concentration due to the wire and discrepancies in thickness).

Following are the results after ultrasonic testing. It was noted that no sticking occurred during wet pressing, which was in favor of the observation of starch influence in pressed samples.

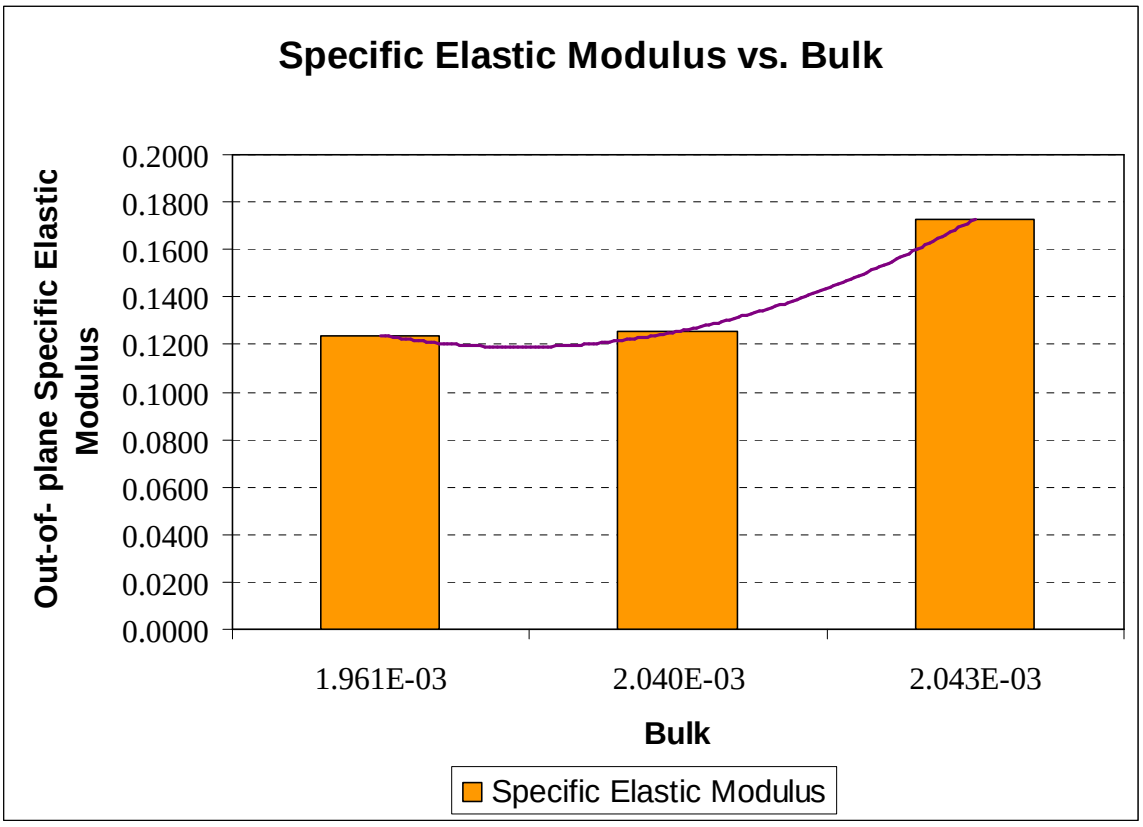


Figure 7: Specific Elastic Modulus vs. Bulk, samples with respectively 0%, 10% and 20% relative content of starch

The consistent trend as compared to the series of impulse dried sample is a good proof of starch integration.

Below are the results in terms of pressure gradient influence, here again under the conditions of wet pressing. The reason for this study again is to grasp in as general manner as possible the potential of high relative content of starch use in the paper industry.

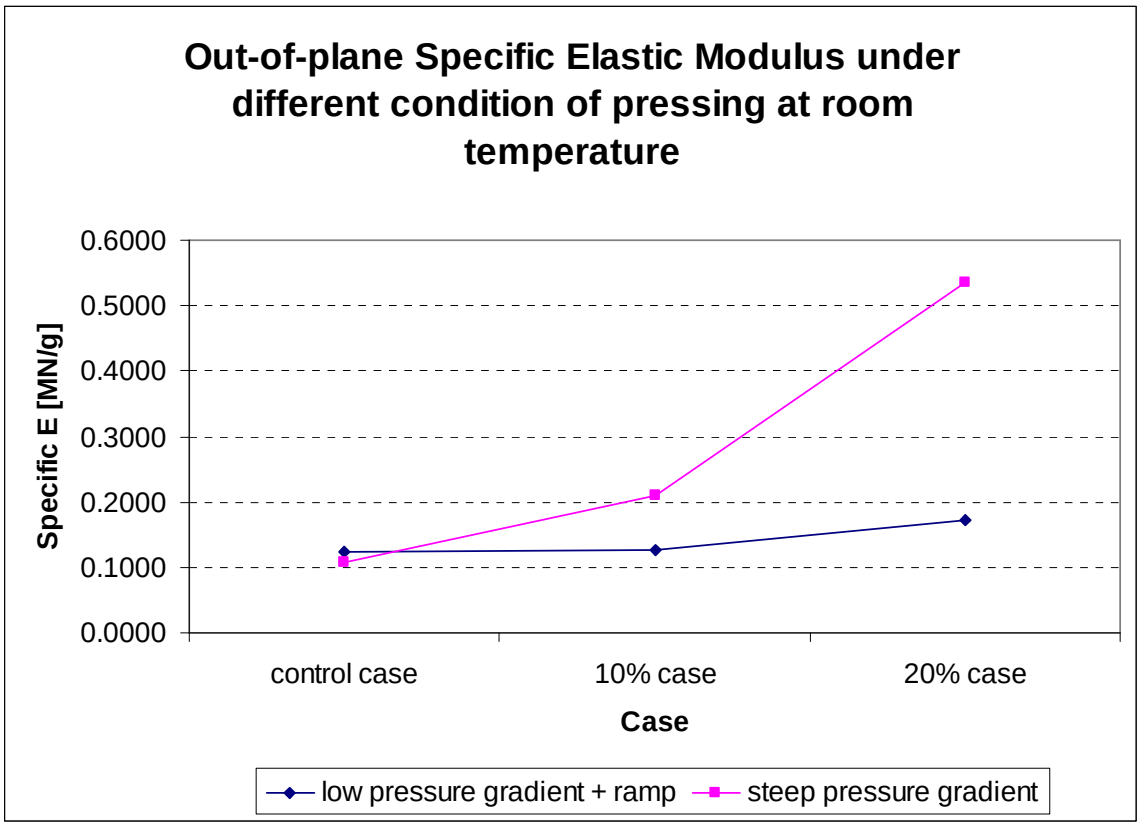


Figure 8: Specific Elastic Modulus for two different pressure profiles

Results concerning specific elastic modulus show the expected trend. In other words, the use of starch in handsheets impacts the overall out-of-plane strength of samples, for any of the two pressure profiles used. While the possible explanation is the amount of energy delivered to the fiber mat during the pressing which differs from the two cases, an investigation on the fiber mat compressibility becomes necessary at this stage along with the formulation of a rheological model.

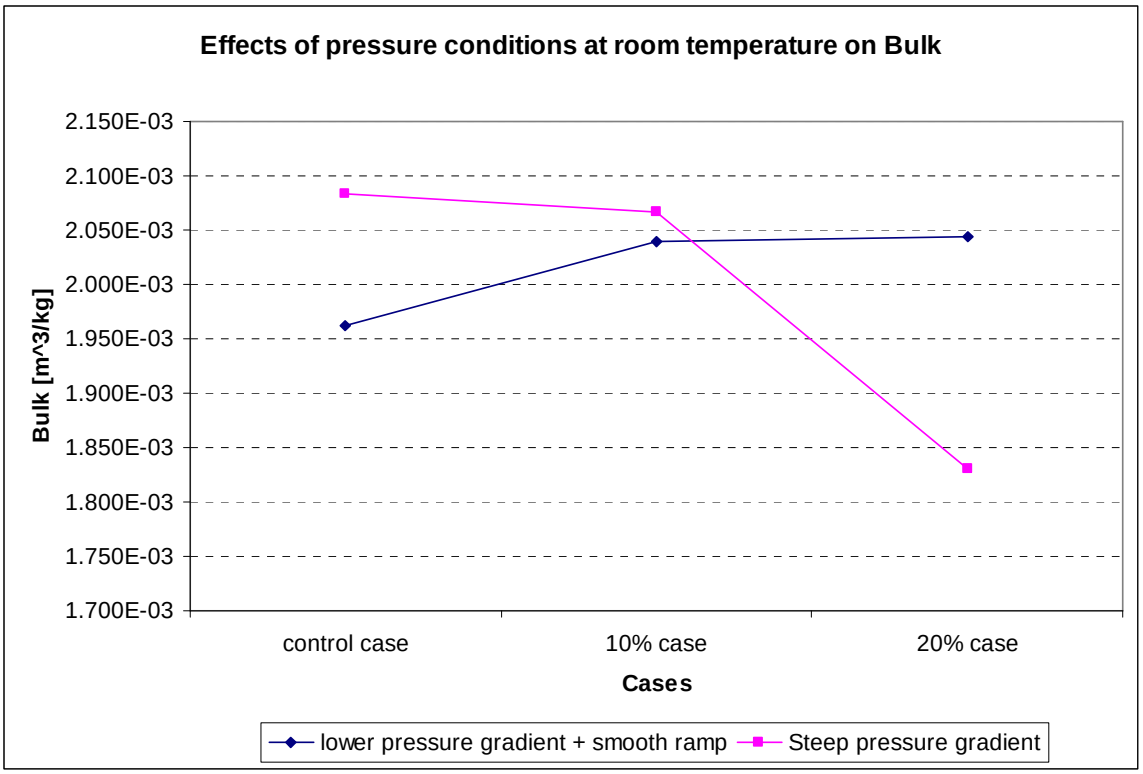


Figure 9: Bulking effect for two different pressure profiles

Interestingly, the results concerning bulking effect on the paper structure show some major differences on the impact of pressure profiles. However, this study still supports the fact that starch has the potential to expand under high temperatures. The reason supporting this hypothesis here is the fact that a sharp pressure release impacts the sheet density as expected, while impulse drying results were showing a puffing effect hence the role of temperature on the starch behavior in the porous structure. Starch alone doesn't seem to impact the bulk of the pressed handsheets while it clearly improves the out-of-plane strength.

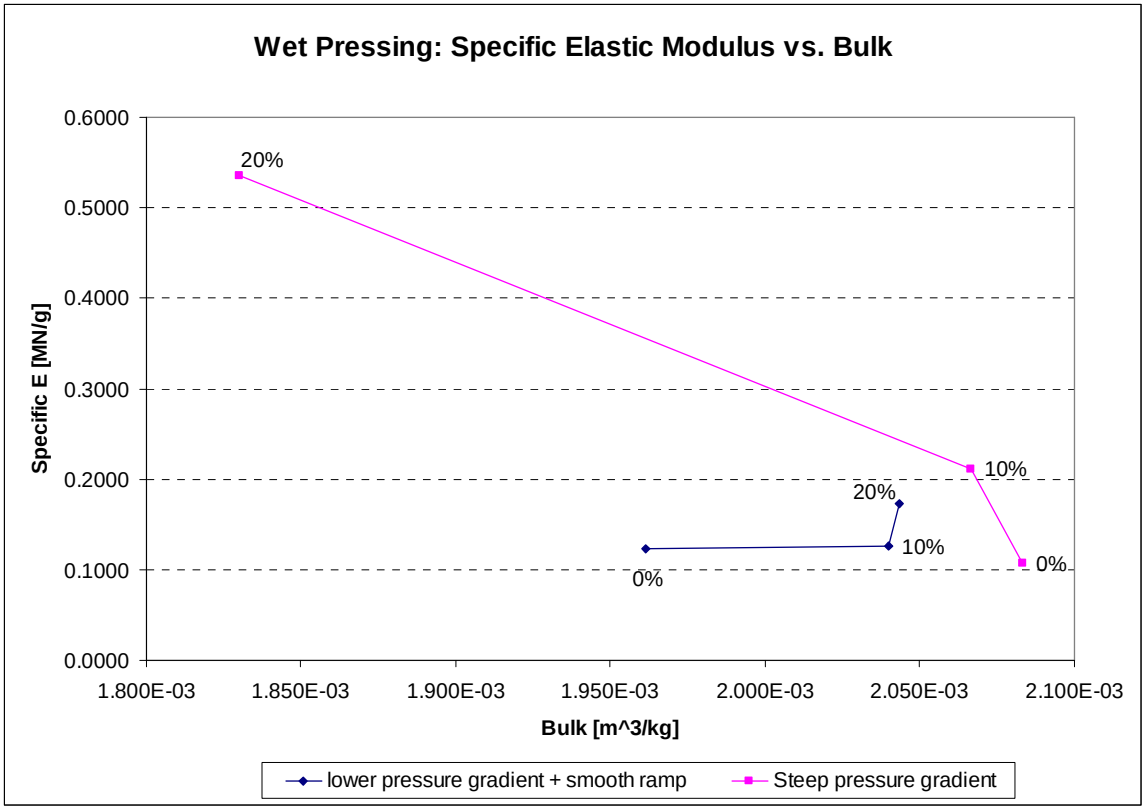


Figure 10: Elastic modulus vs. Bulk for two different pressure profiles

Summarizing the results by showing out-of-plane specific elastic modulus versus bulk gives an interesting perspective for any researcher willing to transfer the use of starch to an increased level of importance in the existing conditions of paper industry. Depending on the choice of grade, we can see that by choosing a specific pressure profile, one can impact the product quality by either enhancing the sheet strength while choosing a highly dense paper bulk, or improving slightly the sheet strength while preserving a reasonable paper bulk. These observations have their importance in terms of energy consumptions in the drying section. One can not ignore the difficulty to maintain a defined pressure profile during the entire process of drying.

Uniformity of Starch Integration

A short series of experiments have been performed in Spring 2007 to try to measure the uniformity of starch integration in handsheet preparation. For this purpose, wet handsheets were tested with a solution of iodide which reacts with starch in the following way:

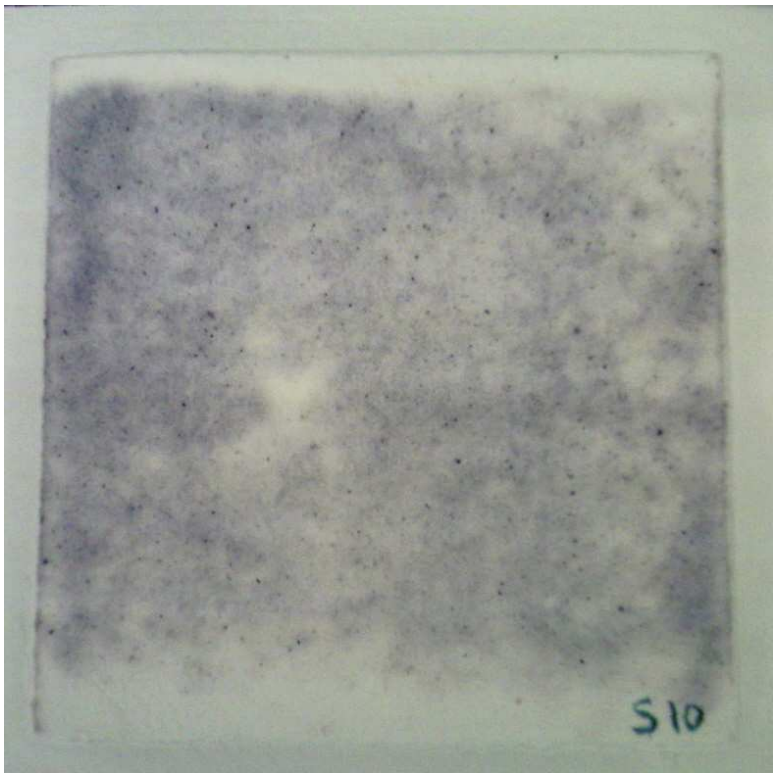


Illustration 13: 10% relative content of starch, handsheet with iodide

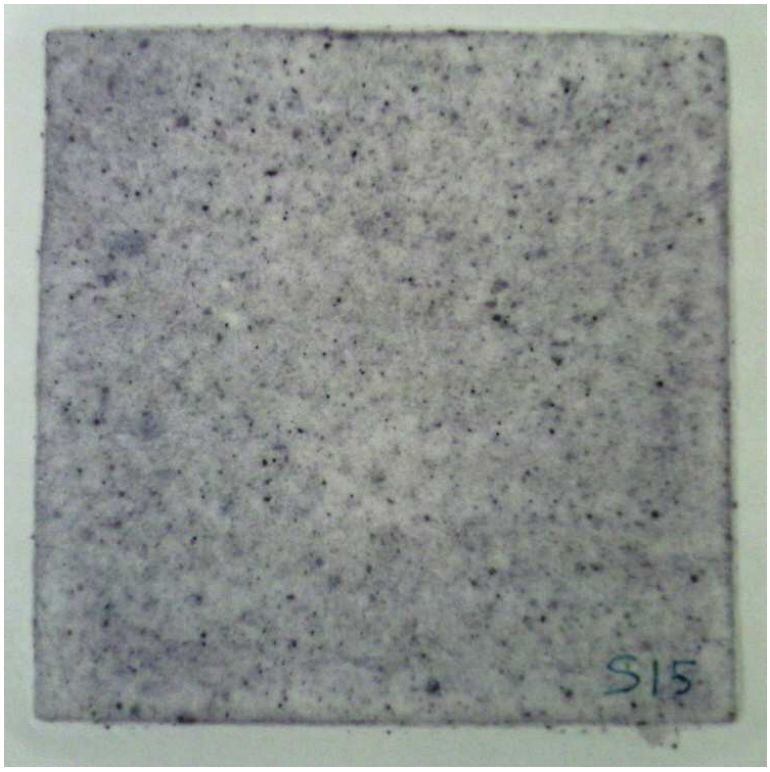


Illustration 14: 20% relative content of starch, handsheet with iodide

While this series of experiments was not quantitative, it interestingly shows the relative uniformity. It was also observed using a matrix of iodine dots with various concentrations that this particular method of putting starch in handsheets was relatively homogeneous in the paper web as well as showing characteristics of repeatability.

Experimental Hypothesis and Plan of Experiments

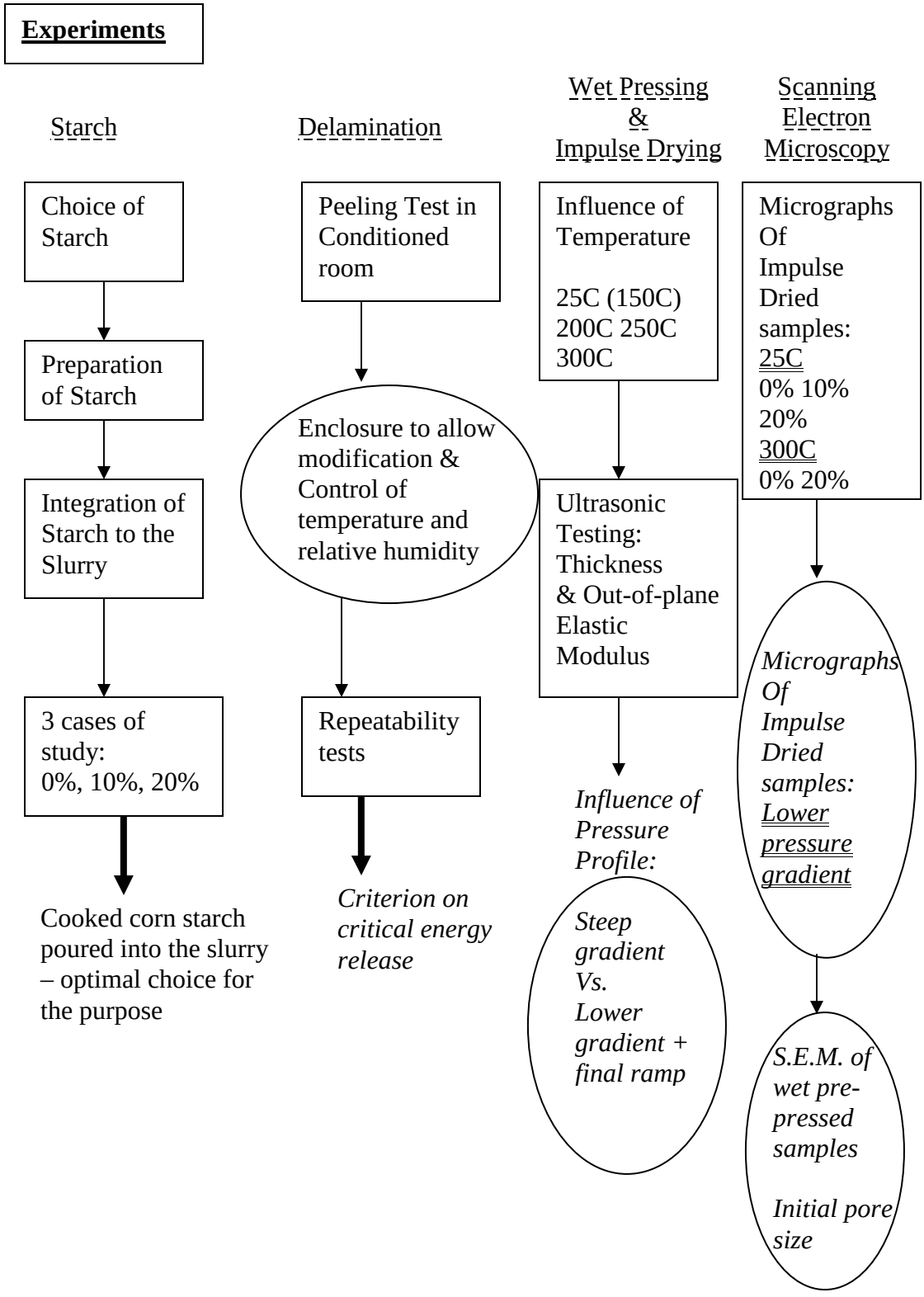
In order to get a better idea on how and why these experiments were conducted, a series of flow chart have been put together.

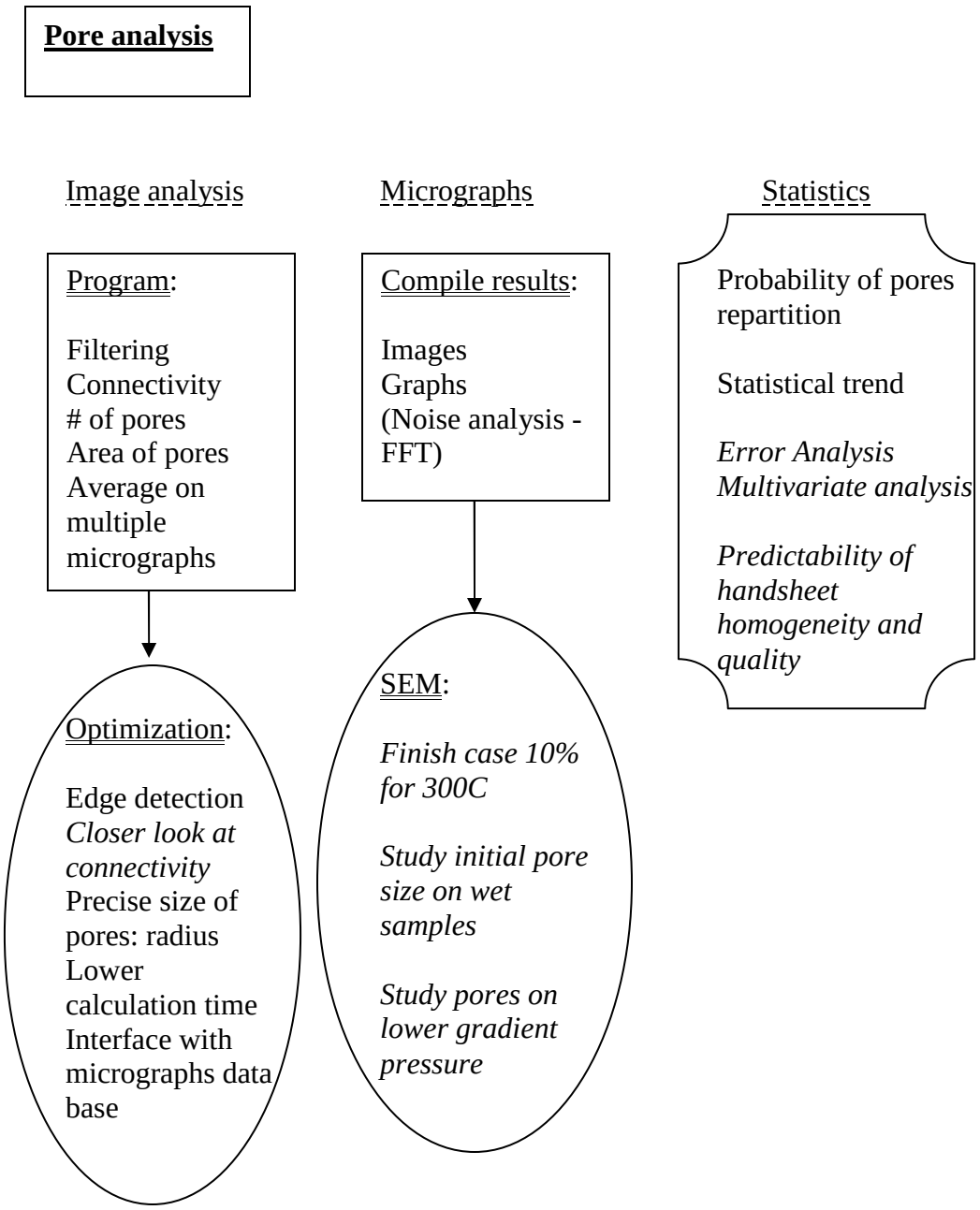
It was essential to work on a hypothesis to guide the research. For this thesis, the idea was to investigate possible new ways of avoiding delamination. At the same time, it was our goal to better understand the influence of impulse drying on the inner structure of paper. With this in mind, it was clear that the pore structure of the wet and dry web needed to be investigated if one desires to explore fiber bonding.
The idea behind was to find something available that would enhance fiber bonding, and would influence the out-of-plane specific elastic modulus. Experiments concerning the "peeling force" of handsheets reinforced with starch lead to the question of bulking and starch behavior under high intensity conditions. Starch can indeed "puff up" under high temperature, and it's only fair to question the gain or loss in strength while there exists a possibility of bulking (which then goes against the densifying effect of impulse drying).

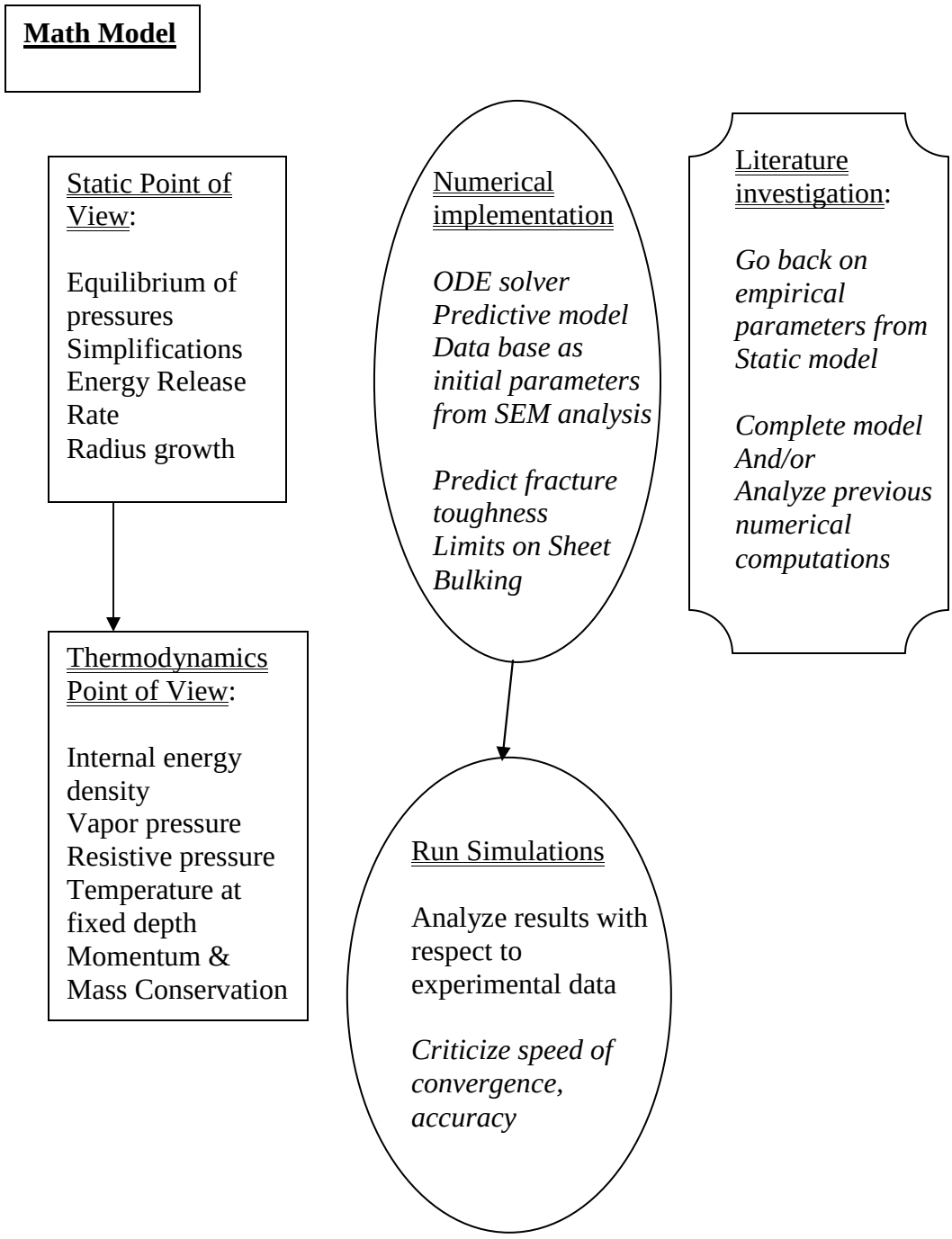
Early studies with handsheets reinforced with a specific choice of starch, impulse dried under a wide range of temperatures have shown promising results in terms of strength and bulk. The logical choice was then to investigate the process of bulking behind the use of starch. For this purpose, it was hypothesized that starch has the potential to close the porous structure of the paper web (at least for small pores), thus encapsulating water without the possibility of venting. While this would greatly increase the risk of delamination (that is by not allowing vapor to vent out the web) the fact that starch enhances the inner strength of the sheet could negate this effect. If this hypothesis is to be verified, there are various parameters to study.

The first concerns the porous structure of the dried web. This is where image analysis is needed, in order to compare the samples in terms of porosity and size of pores.

The following flow charts are to be taken as a general guideline concerning all the parameters to be studied, how the experiments have been conducted and where future work would be done. The steps to be done are indicated in italic.


Experiments
Starch
Delamination
Wet Pressing & Impulse Drying
Scanning Electron Microscopy
Choice of Starch
Preparation of Starch
Integration of Starch to the Slurry
3 cases of study: 0%, 10%, 20%
Cooked corn starch poured into the slurry – optimal choice for the purpose
Peeling Test in Conditioned room
Enclosure to allow modification & Control of temperature and relative humidity
Repeatability tests
Criterion on critical energy release
Influence of Temperature
25C (150C) 200C 250C 300C
Ultrasonic Testing: Thickness & Out-of-plane Elastic Modulus
Influence of Pressure Profile:
Steep gradient Vs. Lower gradient + final ramp
Micrographs Of Impulse Dried samples: 25C 0% 10% 20% 300C 0% 20%
Micrographs Of Impulse Dried samples: Lower pressure gradient
S.E.M. of wet pre-pressed samples
Initial pore size


Pore analysis
Image analysis
Micrographs
Statistics
Program:
Filtering
Connectivity
# of pores
Area of pores
Average on multiple micrographs
Compile results:
Images
Graphs
(Noise analysis - FFT)
Probability of pores repartition
Statistical trend
Error Analysis
Multivariate analysis
Predictability of handsheet homogeneity and quality
Optimization:
Edge detection
Closer look at connectivity
Precise size of pores: radius
Lower calculation time
Interface with micrographs data base
SEM:
Finish case 10% for 300C
Study initial pore size on wet samples
Study pores on lower gradient pressure


Math Model
Static Point of View:
Equilibrium of pressures
Simplifications
Energy Release Rate
Radius growth
Thermodynamics Point of View:
Internal energy density
Vapor pressure
Resistive pressure
Temperature at fixed depth
Momentum & Mass Conservation
Numerical implementation
ODE solver
Predictive model
Data base as initial parameters from SEM analysis
Predict fracture toughness
Limits on Sheet Bulking
Literature investigation:
Go back on empirical parameters from Static model
Complete model
And/or
Analyze previous numerical computations
Run Simulations
Analyze results with respect to experimental data
Criticize speed of convergence, accuracy

Porosity and Mass Transport

Tortuosity

Further pore analysis of viscoelastic materials (paper)

Information on internal phenomenon

Thermodynamics of porous materials

Multiphase phenomenon

Temperature profile

Flow in porous media

Possibility of flash evaporation in a closed pores matrix

Theoretical model of pore expansion

Complete and improve based on previous porous materials study (e.g. transport in bones, vulcanology, and geology)

Non intrusive pore analysis, as opposed to mercury porosity measurement or image analysis

Polymers: Properties

Study on Starch

Empirical values
Main properties
Statistics on bonding strength and ability

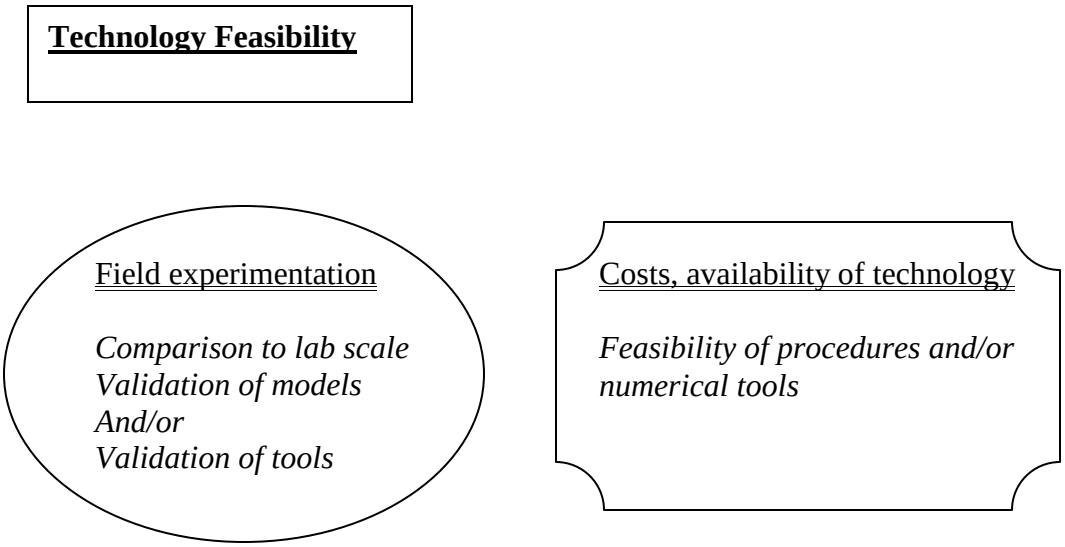
Other Polymers

Similar properties
Cost, Usability
Strength Agent

Mixed properties

Cooked starch with uncooked starch (retention, behavior)

Cooked starch with baking soda (sodium bicarbonate)



Future Work – Experimental Difficulties

Influence of Pressure Gradient at Nip Opening

This section is intended as a guideline for future experiments. For various reasons (time, equipment and funding), more extensive experiments were not performed on the relevance of the pressure pulse shape at nip opening. It was hypothesized that the pressure gradient would have an impact on both pore closing and the bulking effect. The combination of high temperature and pressure indeed has an impact on polymers in general, and especially on starch for its capability of expanding. Since the starch seems to be uniformly integrated to the paper web, it is expected that small pores should be closed under the influence of high temperature while flash evaporation occurs at the nip opening. Both effects should block water in these pores and trigger bulking in the hypothesized way. If that is the case, different bulking effects should be observable on both ultrasonic testing and image analysis, depending on the choice of pressure gradient.

Below is the matrix of experiments that were to be run for this study:

Experiments to be Performed – Influence of Pressure

- ✓ 24 samples for the study of wet samples (goal is to observe these samples on the SEM and to determine the original size of pores before pressing).
- ✓ 24 samples for the study of conventional pressing: wet pressing at room temperature, then observation on the SEM.
- ✓ 24 samples to study the influence of pressure gradient on pores and delamination, pressing at 300°C.
- ✓

Table 3: List of experiments to be performed

#1	Study of initial pore size	Preparation of 24 samples for SEM observation. Impulse Drying at 300C after.
#2	Study of Conventional Pressing	24 samples wet pressed at room temperature for SEM observation.
#3	Impulse Drying at 300°C and new pressure profile	24 samples to correlate pores expansion and lower pressure gradient.

Experiments sets # 1 and 2 were intended to analyze pore structures in each cases: wet pressing at room temperature using previous pressure profile, then use the rest of samples on MTS to compare results in terms of Bulk and Specific Elastic Modulus evolution.

Experiment # 3 was to be complementary to experiment from November 2006 in order to investigate the influence of a different pressure profile with respect to Bulk and specific Elastic Modulus. Wet pressed and impulse dried samples will then be observed on the SEM for further pore analysis.

The most important difficulty encountered is the sticking of starch imbued handsheets to the hot platen. On an experimental point of view, it has forced us to use two different experimental procedures. The first procedure was the use of a ply of tissue between the sheet and hot platen, which modifies the overall structure and surface properties.
An alternate procedure was to design a wire mesh that would fit on top of the handsheets in order to help the release at nip opening. This in turn has created the problem of surface modification as well, with stress concentration areas where the wires were pressed against the handsheets.

CHAPTER 4

3D STRUCTURE OF PAPER – POROSITY ANALYSIS

Dealing with the structure of paper is not an easy task. It is therefore essential to define properly the medium studied to assess correctly the study of impulse drying. As a necessary step before image analysis, one has to define the key parameters of porous medium.

Interest on Venting, Pressing and Drying

Impulse drying has been thoroughly studied in order to understand the drying mechanisms that lay behind this technology. Throughout a series of experiments, several insights have been gained to overcome the problem of delamination. In the current work, the idea to further improve the inner structure of a paper web using starch has been investigated. Besides reinforcing fibers bonding, it is desirable to increase the bulk of the paper sheet as much as possible. These desired qualities are influenced by the wet structure of the web, as well as the components of fibers (cellulose, hemicelluloses, lignin…). Early studies on impulse drying have suggested that the non-uniformities of the web are influencing paper properties like permeability. In other words, the way the grade is chosen, and the paper is prepared (refining, basis weight, furnish…) is a key to influencing as well impulse drying performance, and issues on paper structures.

(*In the following, please refer to reference [8] for more information*)

As a general observation, Dr. Orloff has suggested that because of the impulse dried sheet's greater strength – derived from the sheet's higher density – lower quality and cheaper furnishes, such as recycled fiber, may be used in conjunction with reduced

refining energy. This observation points out the importance of studying the key parameters that defines the paper structure, from the fibers to the porous web.

Influence of Basis Weight

It is believed that delamination does not constitute a big issue when dealing with low basis weight grades (45-90 g/m²) hence the study in this thesis of high basis weight grades (250 g/m²). A possible reason for this basis weight effect is that steam can vent out more easily in lower basis weight webs which retain less short fibers (and fines) than higher basis weight webs.

Sheet Permeability

Another series of experiments performed by Dr. Orloff led him to the conclusion that manipulating the fiber and network properties would allow easy escape of flashed vapor to prevent delamination. The idea is to play on parameters such as basis weight, ingoing solids and degree of refining in order to increase permeability and venting. Measurement of permeability is a good indicator of impulse drying performance since it's characterizing the sheet internal structure to flow properties.

Refining and Hydrodynamic Specific Surface

To summarize, refining effects the fibers by damaging the external layers of the latter. This causes fibrillation (as well as creating fines) and cutting. This has the advantage to increase the fiber bonding area, while preserving the fiber strength (mainly defined by the S2 layer) when refining is not pushed to far. As a consequence, hydrodynamic surface of the sheets is almost a constant for unrefined fibers, for any basis weight chosen. Concurrently, the specific surface is increased with the use of refined fibers. The permeability of the sheet is accordingly influenced by the choice of basis weight as described previously.

Macropores

On a structural point of view, the hydrodynamic specific surface and choice of basis weight are parameters related to the notion of macropores as defined by J.D. Lindsay (*In the following, please refer to reference [9] for more information*)

Macropores are considered to be the boundaries between fiber aggregates that pass through the entire Z-direction of the sheet for a low caliper, low basis weight web. When dealing with a higher basis weight sheet, macropores become an average value defined as specific surface development. Macropores no longer cross the entire thickness of the sheet in high basis weight web.

At lower basis weight, permeability seems to be controlled by macropores, since low specific surface is stimulated. The porous structure throughout these parameters is directly influencing the mode of fluid transport during dewatering as well as drying.

On a performance level, this means a higher critical temperature and outgoing sheet dryness due to the fact that larger quantities of fluid are transported through macropores.

Methods of Analysis – Parameters of Study

Physical Characterization of Porous Structure

(*In the following, please refer to reference [10] for more information*)

Various techniques are available to observe the paper web structure. These visualization tools offer various advantages and disadvantages. A common criterion to differentiate them is to consider whether or not these methods are intrusive (or invasive) or not. Another parameter is the capability to produce 2D or 3D visualization. As it will be shown later on, 2D techniques of visualization can be optical microscopy, scanning electron microscopy as opposed to 3D tools such as laser scanning confocal microscopy (for surface characterization) or X-ray Micro Tomography.

In terms of invasive methods that are not visualization tools, TAPPI procedures exist to normalize the use of Gurley porosimetry or mercury intrusion porosimetry. It is of course limitating to use non-visualization techniques, but they offer the advantage of usage simplicity and can provide valuable information on the porous structure of the studied medium.
(*In the following, please refer to reference [11] for more information*)

A conventional way to determine total pore volume in laboratory is based on the difference of two calculated specific volumes (reciprocal density). On one hand, the internal pore volume is defined as the difference between reciprocals of real density and particle density; on the other hand, the intergranular (or void) volume is defined as the difference between the reciprocals of bulk density and particle density. Hence, the sum of pore and void volumes is the difference between the reciprocals of bulk and real densities. The total internal pore volume can be calculated for example by the so called pycnometric method using water and mercury as the displacements liquids.

Interest on Pore Expansion – Issues in Delamination

Now that the big picture around impulse drying and porous materials is defined, it is essential to break down the various issues encountered. The ultimate goal is of course to integrate the various approaches in a whole meaningful project. That is at least how this thesis has been approached and is intended: a methodological tool for future work.
So far, the purpose for impulse drying improvement has been detailed, and the potential of starch reinforced paper web has been approached through a series of experiments. To integrate these concepts in a logical way, it is important to assess the gains and feasibility of such approach.
Obviously the porous structure of paper combined with starch is modified, and it is important to analyze the potential laying behind. Some ideas have been suggested

throughout the various models exposed in this thesis. They certainly don't fit the exact and whole drying process yet, but it is now a fair assumption to say that acting on the porous structure of paper is the major step to take in the future. It is surely a key parameter in transport phenomenon, either with regards to heat or mass transfer, but also a key influence on impulse drying performance, with regards to the liquid displacement thanks to a two-phase flow which evidently affects flash evaporation impacts and possibly rewet reduction.

On Pore-Size Distribution

A few questions should then be addressed, whether on the vapor front propagation (with its evaporation/condensation phenomenon) or the densification process. Our main concern here is the problem of delamination of course, hence the question of bulking due to internal pressurization of the web. Behind this question, one has again to feel concern with some more parameters concerning the porous structure of paper. Among these parameters, and influencing the entire process of drying, is the pore size distribution, with the question of connectivity between pores, as well as capillarity.

(*In the following, please refer to reference [11] for more information*)

The knowledge of pore size distribution is of importance on diffusion rates as already discussed earlier concerning the performance of impulse drying. The concept of macropores has been defined early in the study of impulse drying impact on a paper web. It is therefore important to determine the pores in a given size range and its contribution to the process of drying. Early classifications were using pore radii as a parameter to differentiate micropores from macropores. Hence, pores having a radius lower than 100Å were considered as micropores, and above as macropores.

As described by Gary Rudemiller et Al., the pore-size distribution has its importance in the late stages of impulse drying. It's been observed that the heat flux decays rapidly to reach a quasi-steady state until nip pressure is relieved.

We know that this stage corresponds to the flash evaporation phenomenon, which impacts the inner sheet structure by possible delamination. As stated earlier also, it's believed that delamination is not only controlled by flash evaporation but also by the position of the two-phase flow front in the web. With the experiments on boiling heat transfer mechanisms in impulse drying, Rudemiller has suggested that the phenomenon occurring in the latter part of impulse drying might be controlled by an interaction of sheet pore size, pressure buildup within the sheet, and the so-called counterpercolation of steam and liquid water in the sheet. It is demonstrated that during the iso-heat-flux regime, the heat flux is independent of thermal driving force, but depends on the pore-size of the medium, hence the importance of pore-size distribution. The key of the paper web during impulse drying is its capability of delivering liquid to the heater surface and also the system pressure. It is fairly believed that by the time the quasi-steady regime is reached, free water at the heater is likely used up and the plane of evaporation way within the sheet structure. Thus, the heat flux is controlled by the rate at which the pore structure can provide liquid water to the plane of evaporation for phase change. In this regime, a nearly isothermal zone delivers liquid to the plane of evaporation while venting out the vapor. Hence, the pore-size distribution plays a major role in the later part of impulse drying.

For this question of distribution, it is inevitable to consider a stochastic approach. Besides some experimental test, a numerical model is essential to assess correctly the influence of pore-size distribution on various transport properties. It is possible to get some good indication of various phenomena when observing the later regime's heat flux.

Decrease in this regime could be a proof of continued sheet compression, thus a decrease in pore size of the sheet. Concurrently, a sudden change could be a proof of delamination.

However, to complement this study, as well as double check the results, image analysis comes in handy. Rather than digging into multivariate analysis and defining exotic distribution functions based on porous volume differentiation, working on image samples of the inner structure of the paper web can provide useful data for a further study on pore-size distribution.

For this purpose, a model of pore expansion has been defined to relate pore-size distribution with pore expansion after impulse drying, specifically defined for starch imbued handsheets.

On Pore Connectivity

As important as pore-size distribution, there is also the question of pore connectivity to address before going to image analysis. Actually, pore connectivity is merely an extension to the notion of pore-size distribution. Since a paper web can be seen as a wiring of cellulose fibers, a single pore is potentially connected to its neighboring pores. The question then is how to define such connectivity and differentiate it from micropores, and relay this notion to the connectivity to the surface of the web, which is crucial in terms of venting capability. As we'll see later on, it is actually easier to define a connectivity criterion based on the idea of capillarity in 2D visualization rather than 3D visualization.

In our case, to support or reject the hypothesis of closed matrix with starch reinforced paper, and its influence on delamination; we're more concern by a comparison criterion. Thus, an arbitrary parameter of connectivity can be designed for our purpose, as long as it's used in every study afterwards. As one can see, it is no simple task to accurately

define a connectivity criterion, and this question will be re-addressed during the image analysis part.

CHAPTER 5

APPLICATION IN IMAGE ANALYSIS

2D Against 3D Tools

Great deals of efforts are being made these last years in providing efficient 3D reconstruction of various porous medium. While computer memory capacities and processing speed have greatly improved to render 3D computation affordable, there remain a few problems that still limit the use of real 3D analysis. It is therefore essential to pursue 2D analysis improvements while techniques are transitioning to real 3D computation and visualization.

Data Acquisition

Various techniques of image acquisition are available for the purpose of porous structure analysis. Each has its advantages and inconvenient, depending on the purpose of the study and the parameters of the considered medium. A review of these tools is an important step in approaching digital acquisition. The first criterion to choose a method more than another is to balance destructive to non destructive methods. A non destructive method generally does not cause any changes in the porous structure during the imaging process (X-ray microtomography, confocal laser scanning microscopy, Magnetic Resonance Imaging, ultrasonic microscopy…).

(*In the following, please refer to reference [10] and [12] for more information*)

Light Microscopy

This optical system is able to capture a fairly good range of opacity through thin samples; however, it can introduce geometrical distortion that can be an issue when dealing with stacks of information.

Confocal microscopes are a special type of light microscopes that has an optical slicing capability. The lens system uses a pinhole to create a very thin focal plane which allows the possibility to collect light at a define depth in the sample. Dr. Hiroki Nanko has been one of the first to describe an application of confocal laser scanning microscopy on paper materials, in a study on interfiber bonding in 1989. (Re. [13])
The main limitation of this method is that the signal intensity decreases rapidly with depth of observation, due to the refraction of the beam with fiber layers.

X-Ray micro tomography

Tomography offers the advantage to locate boundaries between fibers and pores. There are two principles concerning the process of microtomography: beam absorption or phase contrast. Since paper is made of carbohydrates and lignin, fibers is a weakly absorbing medium, so phase contrast is the most adapted technique of visualization in this case. Phase contrast is efficient in regions of sharp changes in the refractive index of the sample, as it's the case on the borders between fibers and pores. However, some issues exist concerning the creation of artefacts that have to be addressed with a particular attention. The technology to create efficient X-ray beams is also quite demanding, and requires synchrotron sources that are not found in everyday labs. However, its resolution capacity is definitively a plus; recently Holmstad et Al. obtained a resolution down to 0.7 μm^3.
(*In the following, please refer to reference [14] for more information*)

Scanning Electron Microscopy

The major tool used for this thesis in terms of image analysis has been scanning electron microscopy. Several microscopes are available at the IPST. They have the great advantage to go down in resolution to far better ranges than those attained by visible light techniques. On a practical point of view, the fact that porosity is the concern in this thesis

allows a reasonable use of the S.E.M. resolution that minimizes the possibility of artefacts and deformation of the micrographs.

The only limitating factor in using the S.E.M. is the necessity to prepare epoxy embedded samples of the specimen to visualize. In order to preserve the paper web structures, it is important to follow a series of treatments, using designated chemicals that won't affect the fibers structures nor bonding.

In this study, the out-of-plane properties are concerned, thus micrographs of the xz/yz plane (transverse structure) were taken as shown below (no MD or CD directions concerned since the handsheets are lab-made)

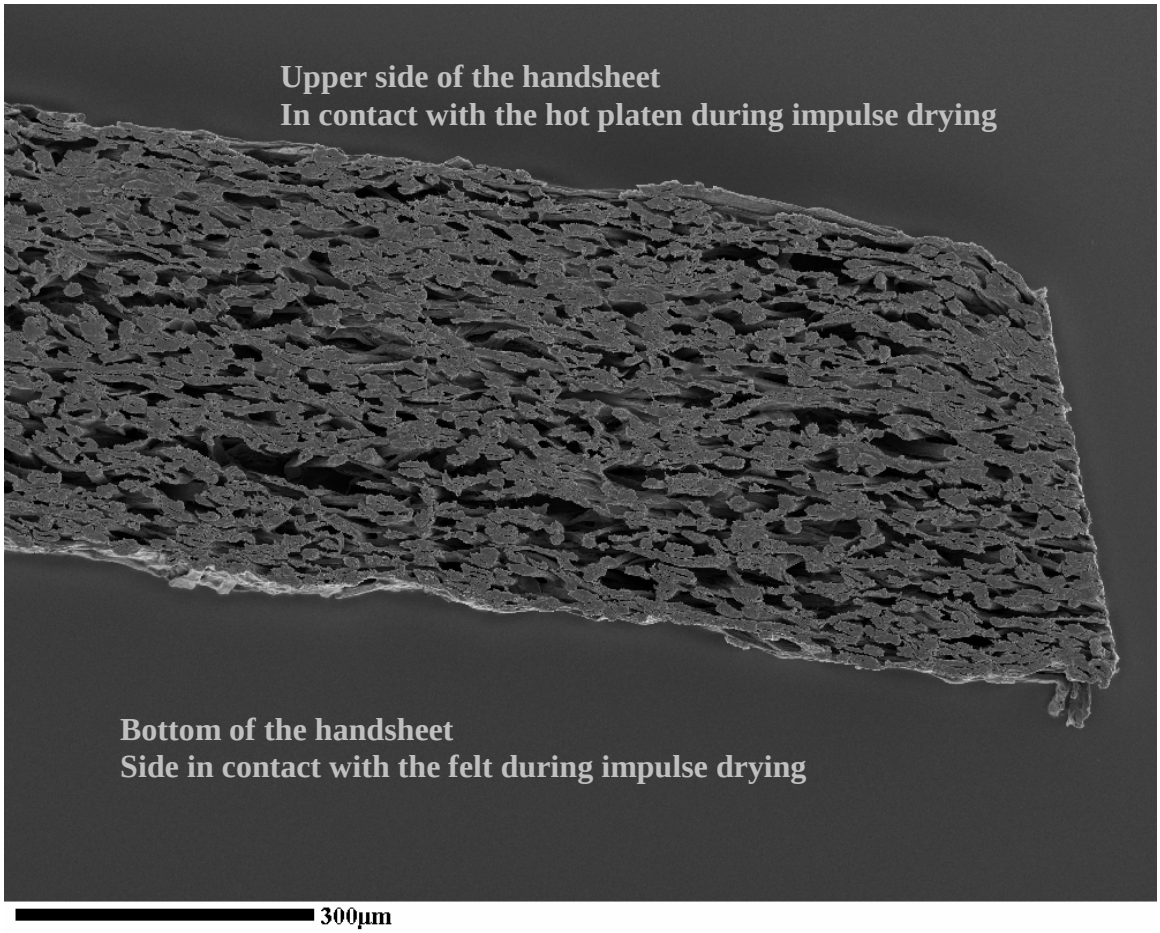


Illustration 15: Starch imbued handsheet with a relative content of 20%, wet pressed at room temperature

Methods

(*In the following, please refer to reference [15] for more information*)

The advantage to use image analysis is the possibility to handle numerical format. Accessing digital information allows various procedures and access to properties that physical testing cannot even approach. Measurements are accessing a wide range of fibers properties, such as fiber analysis, fiber network characteristics (average length, bonds…) and fiber pore network (tortuosity, connectivity…).

The first important step once a digital image has been acquired is to pre-process it in order to enhance its overall quality. It is essential to handle as best material as possible in order to minimize the variety of errors one can encounter while working with computation. Among the parameters to check before computation, noise attenuation, lens corrections of optical system (when such equipment is used), attenuating artefacts from the imaging process are to be consider to adjust the image.
The second step is, as far as porous structures are studied, the determination of the main entities in the image. It is essential to determine the geometrical features of the medium to be studied as a support for further applications in terms of numerical models or physical properties. For this purpose, the adaptation of the objects in a more convenient format is to be considered as well. A major step here is to keep an accurate track of the transformation used in order to back up the properties in the global system studied, as well as to offer the possibility to interpret correctly the measurements. Converting the geometrical structure of a paper web with micrometer resolution can turn out to be a non-trivial task.

Under these regards, sampling a portion of the paper web to be analyzed is a key parameter as the method used for data acquisition. Among the most popular methods,

tomography and sectioning by microtome cutting are the most convenient. A microtome is a high precision cutter, usually equipped with a diamond or glass blade. Some new generation microtomes can cut samples down to 50 nm thick.

However, some simple and practical approaches are also pretty useful when dealing with epoxy embedded samples.

Sample Preparation

Each case of study has 8 different samples which are 4” diameter handsheets. From these samples, two square specimens are taken to be embedded for microscopy observation. The side of each specimen is of exactly 2 cm in order to keep coherent and preserve the reproducibility of the experiments.

Illustration 16: Coated and Uncoated samples. Each epoxy sample counts 8 handsheets samples.

A particular attention has to be put on preparing samples for embedding. Materials used for sample embedding are different types of epoxy resins or glycol methacrylates, the hardness may be varied to suit particular paper material.

A particular attention has to be put on the use of chemicals at this stage of the preparation. Not only security is essential, and can never be too much emphasized; but depending on the availability of chemicals, the date of opening of these latter might be a limitating factor leading to poor curing of samples. This will render the observation quite useless as one can see on the following micrograph.

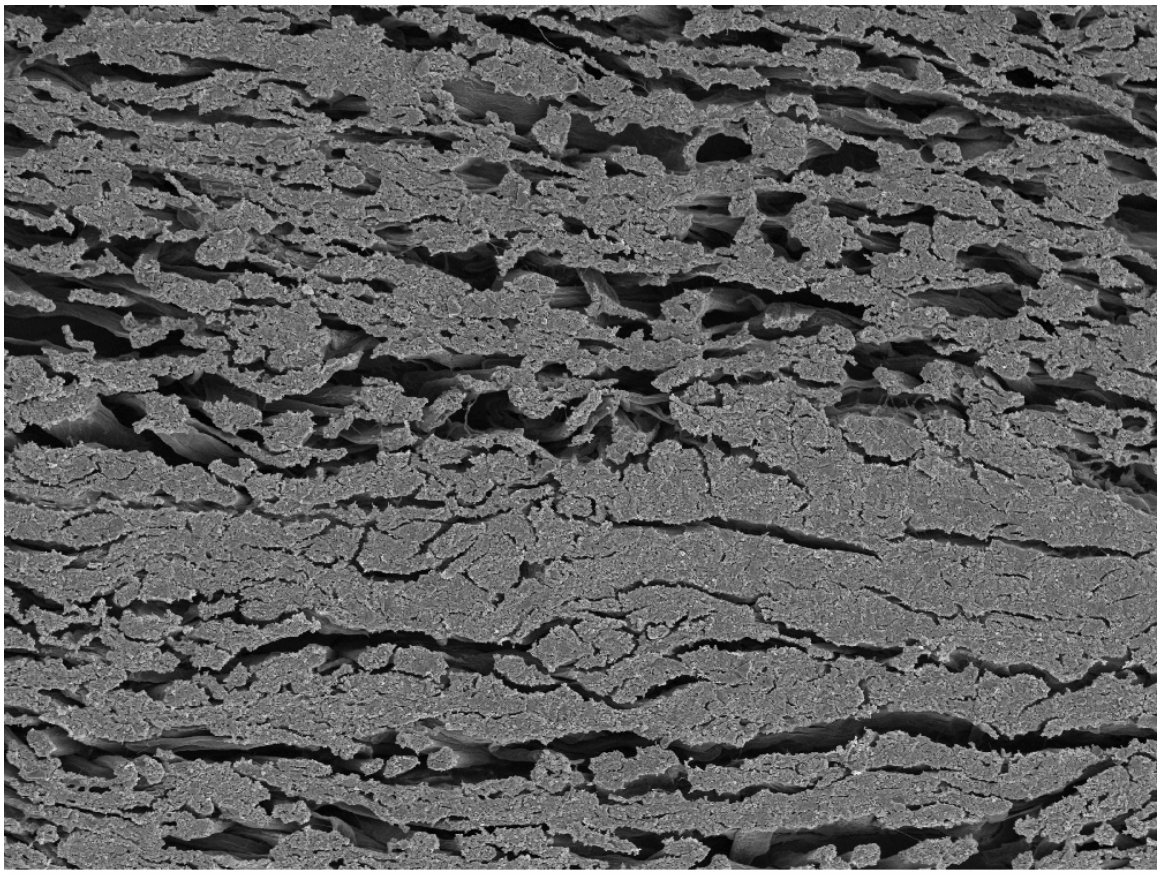


Illustration 17: Example of handsheet surface altered by poor chemicals use

The last key step to electron microscopy observation is gold coating. In order to prevent samples from burning, it is essential to coat them with a conductive layer of metal, usually gold with the desired thickness. Too much coating will affect the visualization considerably, while not enough will lead to rapid burning of samples which is clearly not desirable.

Micrograph convention

The micrographs are all of a size of 750 x 1000 pixels. The resolution of a pixel is recorded after a choice of magnification such that the height of the micrograph includes the entire thickness of the sample. For further studies, the exact position of pores with respect to depth (in the out-of-plane direction) can be easy to determine.

The file is saved as ".TIFF" document which provides the most precise information for our purpose. Micrographs are in black and white, which is to say a range of gray levels between 0 and 255, where 0 stands for black (which mostly stands for pores) while 255 is white (which stands for fibers, fines and starch). Our micrographs are typically 8-bit images, which gives a 6 Mb of necessary storage capacity per micrograph.

As said previously, the goal was to take micrographs on the entire thickness of the samples. The magnifications used for this purpose were always in between x100 and x300, which corresponds to a pixel size of 1.16 µm down to 0.387 µm; except for the sake of specific observation as shown below.

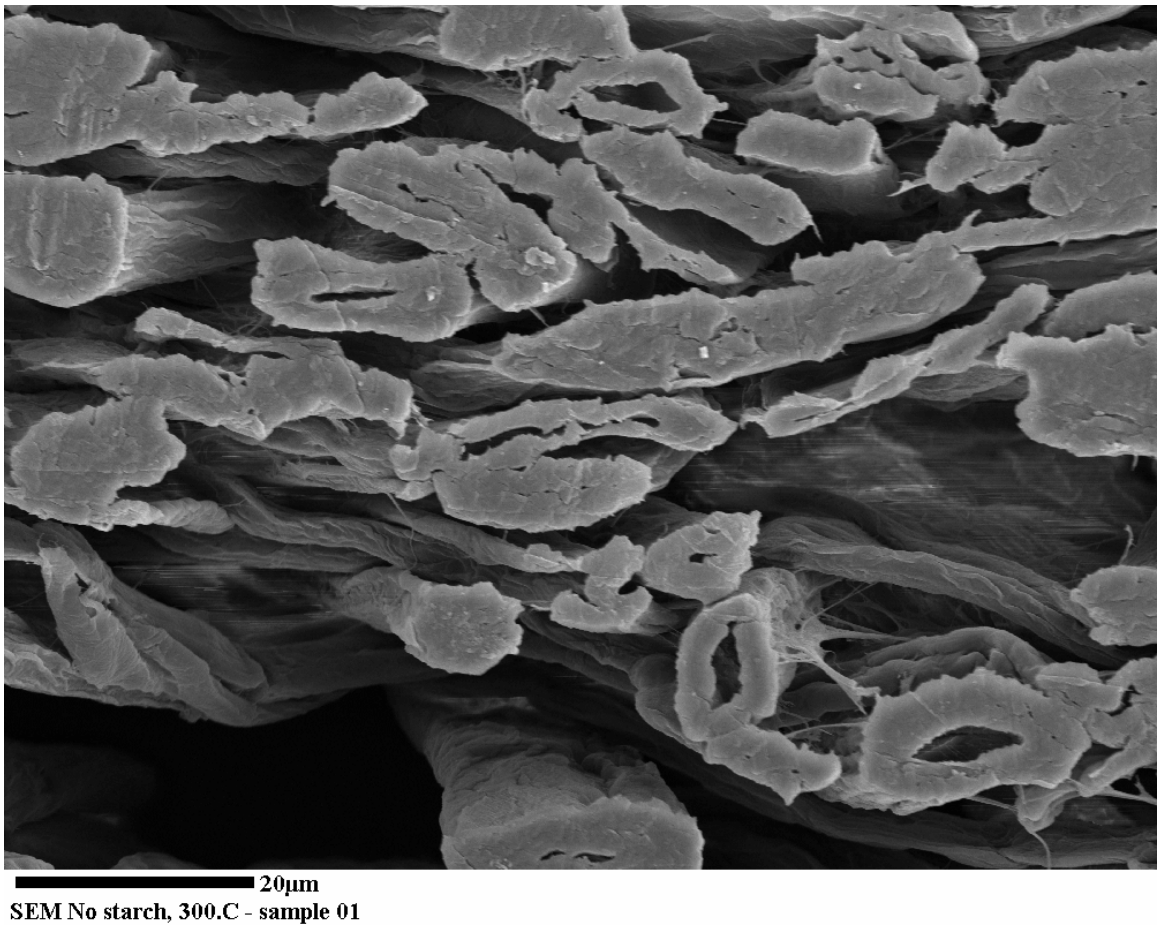


Illustration 18: Impulse dried handsheet at 300°C, no starch. Observation of fiber bonding

The above is at magnification x1200 (1 pixel = 0.097 µm)

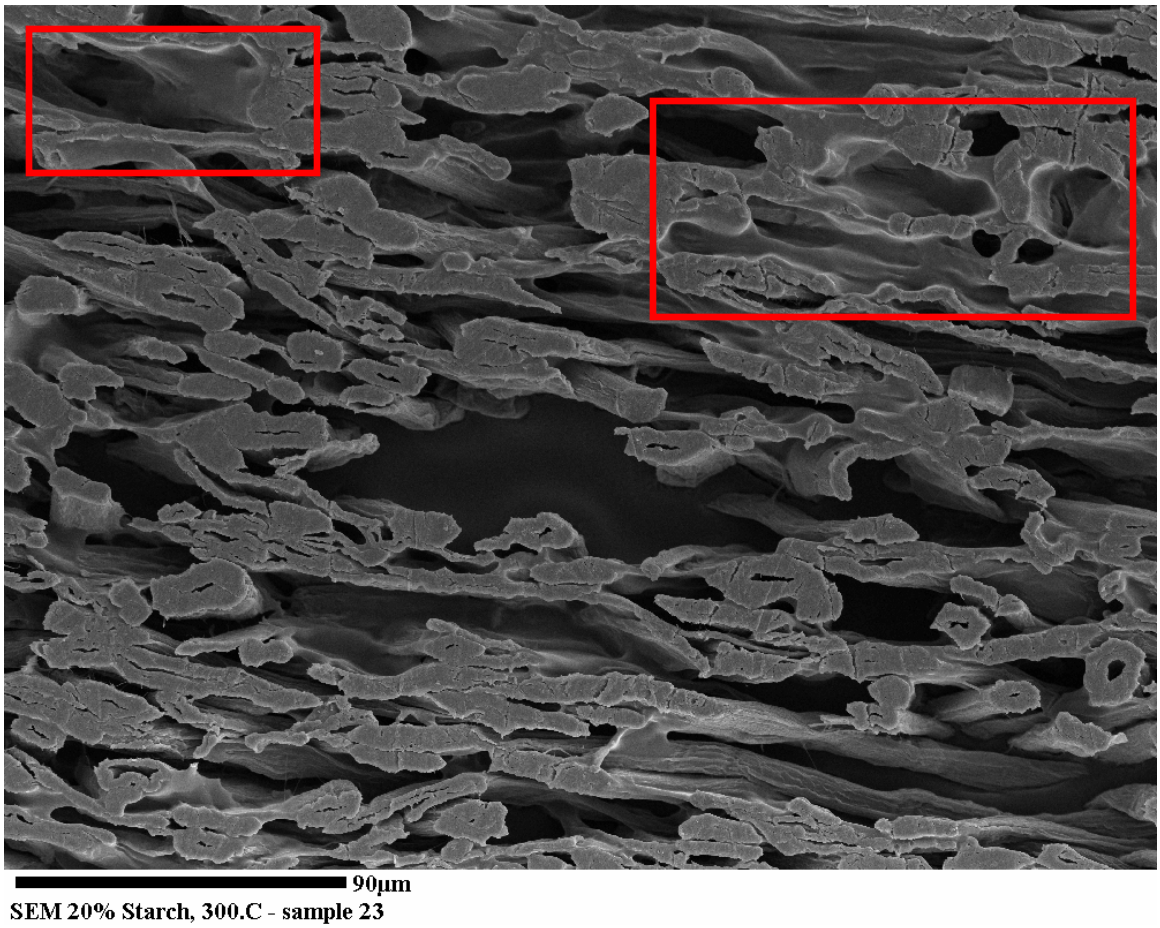


Illustration 19: impulse dried handsheet at 300°C, with a 20% relative content of starch

The above is taken at a magnification of x370 (1 pixel = 0.314 µm) to observe the relative integration of starch on fiber bonding. In this case, it is noticeable that starch has efficiently coated the fiber network, as indicated by the red boxes.

Theory behind Image Analysis

Once a digital support is available for analysis, it is crucial to perform a few alterations to it, depending on the purpose of the study. Generally, a digital image contains various errors that limit at first hand the study; whether it is in terms of resolution (blurry image), contrast, intensity or random noise from background. It is also important to decide whether or not the image range will be used under gray levels representing intensity

levels or under the RGB format (standing for Red-Green-Blue, color representation). For all these reasons, transformation is a first step to image analysis.

Filtering

(*In the following, please refer to reference [16] for more information*)

When dealing with imaging devices (for our case, scanning electron microscopy), the first problem occurring at first observation is the possible noise coming from the background of the image. Artefacts from inhomogeneous background are often encountered and severe problems when dealing with the analysis of such digital support. Automatic processing, such as shape recognition, edge detection, features reconstruction; becomes non-trivial tasks. The natural idea is then to define a filter in order to minimize these undesirable effects. The fact that a digital image is a linear time invariant system helps considerably in this purpose. The signal processing to such a system can easily be expressed as a convolution between the so-called input signal and the system kernel. Hence, a system kernel can be designed as a filter (ideally a Dirac pulse is the best option) and convolution-based techniques for image restoration are the most common tool for reconstruction. Linear models such as iterative blind deconvolution, Wiener-based method or discrete filter mask optimization are common as a first approach to filtering.

(*In the following, please refer to reference [17] for more information*)

When acting on features like the pixels of the image, the process is called spatial filtering to clearly differentiate it from signal processing and its frequency domain filtering. The process consists simply of moving a filter mask from pixel to pixel in an image. At each point (x, y), the response of the filter at that point is calculated using a predefined

relationship. For linear spatial filtering, the response is given by a sum of products of the filter coefficients and the corresponding image pixels in the area spanned by the filter mask.

This technique can be illustrated by the convolution of an image f with a mask w:

Equation 2: Definition of a filtering mask

$$g(x,y) = (f * w)(x,y)$$
$$= \sum_{\substack{(x',y')\in W \\ (x-x',y-y')\in F}} f(x-x',y-y')w(x',y')$$

where

$W \equiv$ *set of position in window*

$F \equiv$ *set of position in image*

$w \equiv$ *mask of weights*

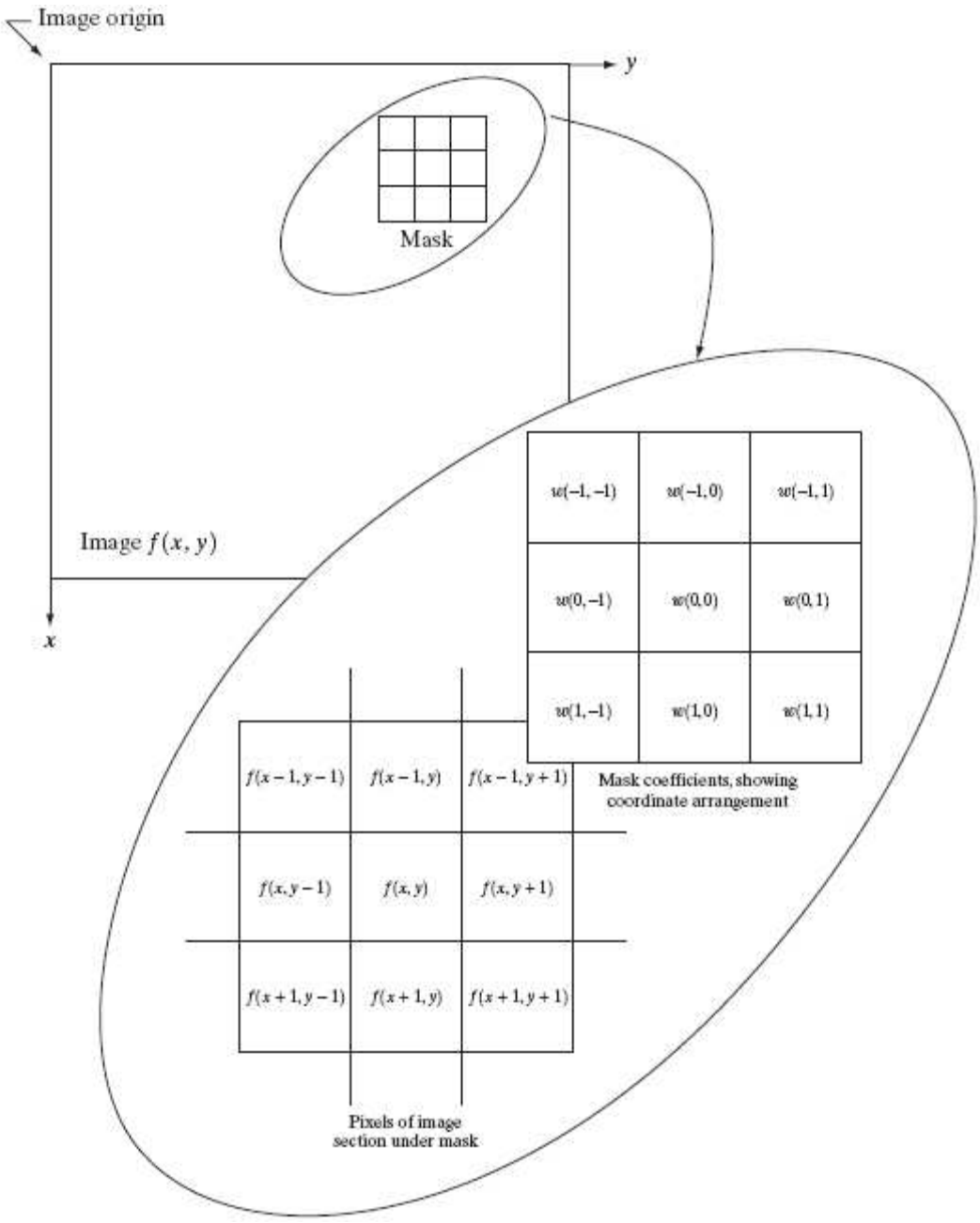


Illustration 20: Example of spatial filtering using a 3x3 mask

There are several types of filter, depending on the desired response. For noise and blurring reduction, these are called smoothing filters. The idea is to reduce sharp transitions in gray level of the image by replacing pixels intensity value by an average of the neighboring values. Since random noise consists usually of sharp transition, this kind

of filter works well, but it also affects edges which are another type of sharp transitions in the image. For our case, this is an undesirable side effect that emphasizes the importance of filter design with respect to the desired study.

As described later on, it is often necessary to combine various filters to obtain the desired result in image transformation. While a median filter avoid edge blurring but minimizes random noise; a Laplace filter enhances intensity variations, but is noise-sensitive due to second order derivatives. Hence, filter design is not a trivial task and a key element to image analysis. Our purpose being geometrical features study, it is necessary to keep as much detail as possible, while minimizing random noise and other parasite effects.

Contrast Enhancing

This is another critical step in image analysis. Contrast enhancement is highly desirable in the study of geometrical features in a digital image. One has to make sure the range of intensities is clearly defined before taking any further steps. As discussed previously, minimizing undesired details and sharpening desired intensities is one thing, but working on the transitional range of intensities is also important and helpful in the differentiation of features.

Contrast enhancing techniques basically work on the intensities histogram of the images which can be visualized by the function *imhist* from Matlab. Since Matlab is used for the image analysis in the context of this thesis, we'll focus from now on specifically on Matlab features, without however loosing the theoretical background.

Matlab © help:

imhist(I) displays a histogram for the image I above a grayscale colorbar. The number of bins in the histogram is specified by the image type. If I is a grayscale image, imhist uses a default value of 256 bins. If I is a binary image, imhist uses two bins.

A first approach is to use the function *histeq* that enhances the contrast by transforming the values in an intensity image, or the values in the *colormap* of an indexed image, so that the histogram of the output image approximately matches a specified histogram. Although the output histogram matches the original histogram, the true nature of the sample seems to be lost by the fact that the entire histogram is shifted in order to fit the entire range of gray values, that is from 0 to 255. This is a problem by the fact that our 2D sample is taken from a 3D fiber structure. This has the disadvantage to reveal fibers in the background that are from a subjective point of view part of a pore on the surface.

It's then preferable to use adaptive techniques such as Contrast-limited adaptive histogram equalization (CLAHE), which is performed by the Matlab function *adapthisteq*:

Matlab © help:

CLAHE operates on small regions in the image called tiles rather than the entire image. Each tile's contrast is enhanced, so that the histogram of the output region approximately matches the histogram specified by the 'Distribution' parameter. The neighboring tiles are then combined using bilinear interpolation to eliminate artificially induced boundaries.

Basic distributions are defined by the following parameters:

Uniform: corresponds to a flat histogram

Rayleigh: corresponds to a bell-shaped histogram

Exponential: corresponds to a curved histogram.

Here again, some distributions are more adapted to our problem than others. Since we're focusing on determining pores in the handsheet out the material part (which can contain

fibers as well as starch), it is important that we keep the original lower values in the intensities histogram as well as making use of the entire range. Thus, the Rayleigh distribution which is somehow shifting the bell-shaped histogram to the right on a centered mean value is not adapted to our case.

Observing the effects on an actual micrograph, we see that in this case again, background fibers are enhanced and appear in the front although they should be hidden inside the pore to account for the surface area of this pore.
This let us the only choice of the exponential distribution, which appears to be exactly what we desire: it keeps the original lower value and smooth the distribution down to the higher value in a continuous manner.

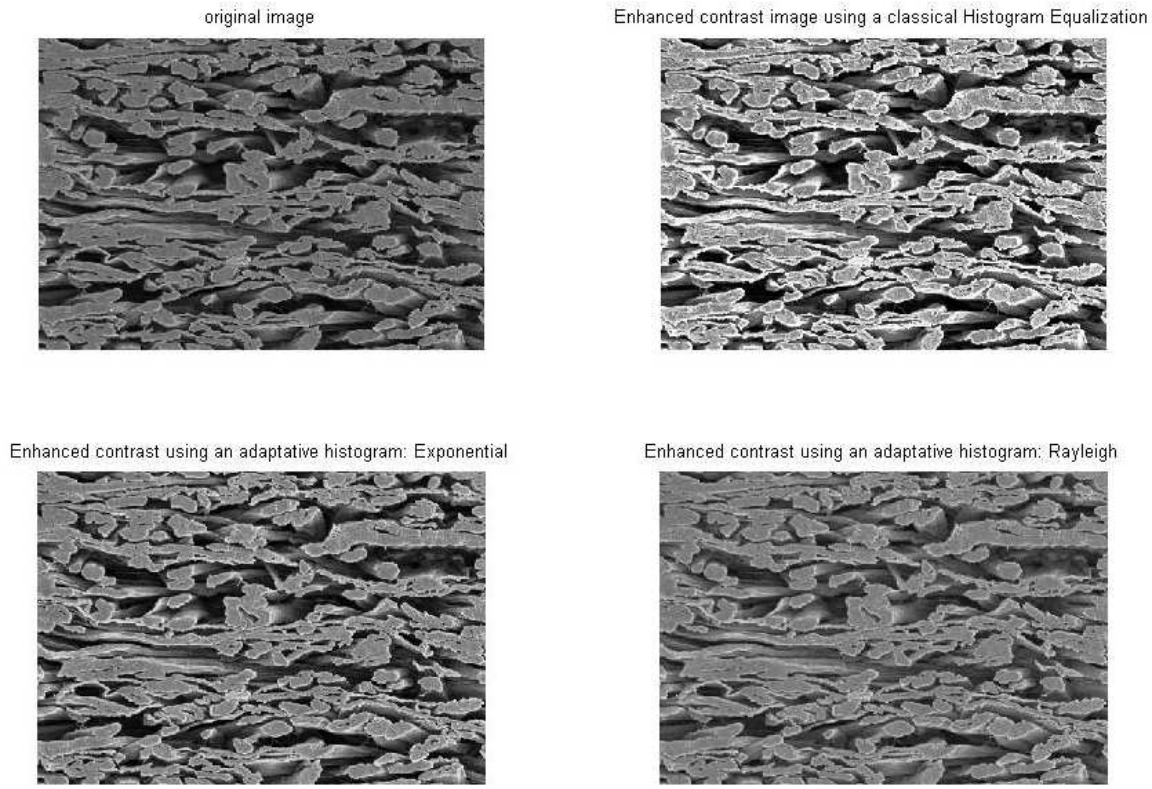


Illustration 21: Comparison of contrast enhancing techniques

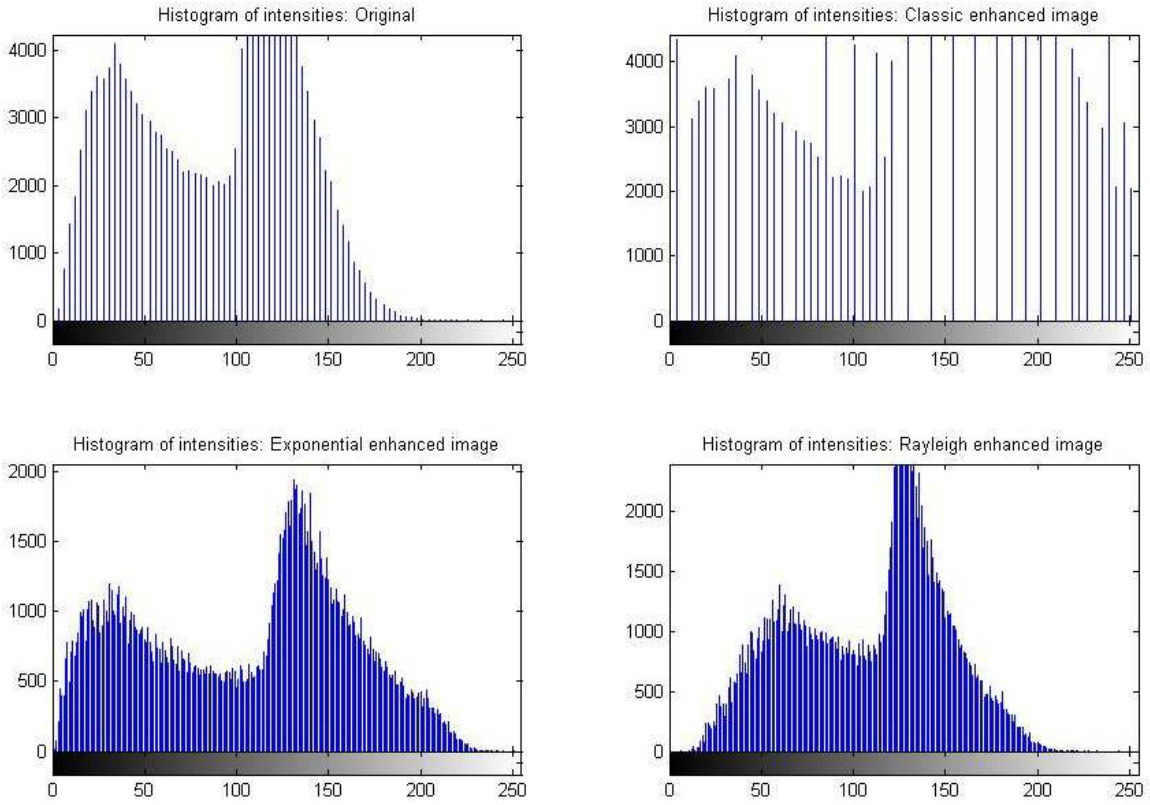


Illustration 22: Typical contrast enhancing results in terms of intensity histograms

As seen above, it is more rigorous to work with intensity histograms rather than judging a priori on the sole impression of the eye. Intensity histogram is an essential tool in image analysis as we'll see later in the application of image analysis to paper study.

As a general observation, it is clear with the intensity histogram that exponential method is adapted to our goal in observing porous structure of paper web. One can observe also the shape of this histogram which is common to all sample, and especially regarding the two maximum values in the lower and upper part of the range. These values will come in handy to apply thresholding to our micrographs.

Binarization – Thresholding

Having improved the intensities histogram, it is a good idea to make use of it to determine a range of thresholds. The idea is that main features in gray-level micrographs can be distinguished by one or more thresholds. Clearly, in the case of paper structure, pores will be featured by the lower range of intensities from 0 to a certain intensity value T1; while fibers will be featured in the upper range of intensities from a value T2 to 255. Hence, ideally two thresholds are needed but they will define an undetermined range from T1 to T2 which will need a particular attention.

It is important here to keep in mind that we're trying to determine pores out of a micrograph which already contains errors. We don't want to increase this error margin, and determining thresholds can be a big source of error in this case, especially in such a complicated sample as a starch imbued handsheet. Working on a 2D sample out a real 3D sample created from a fiber assembly is not an easy task.

Working on a threshold range is for this purpose an advantage. Working on a single threshold like most of the image analysis programs suggest (meaning binarizing the image) induces in our case a big error. We cannot simply binarize our micrograph using only one threshold. There is no such thing here as fibers above one threshold value T*, and pores under that particular threshold value.

Example of Binarization

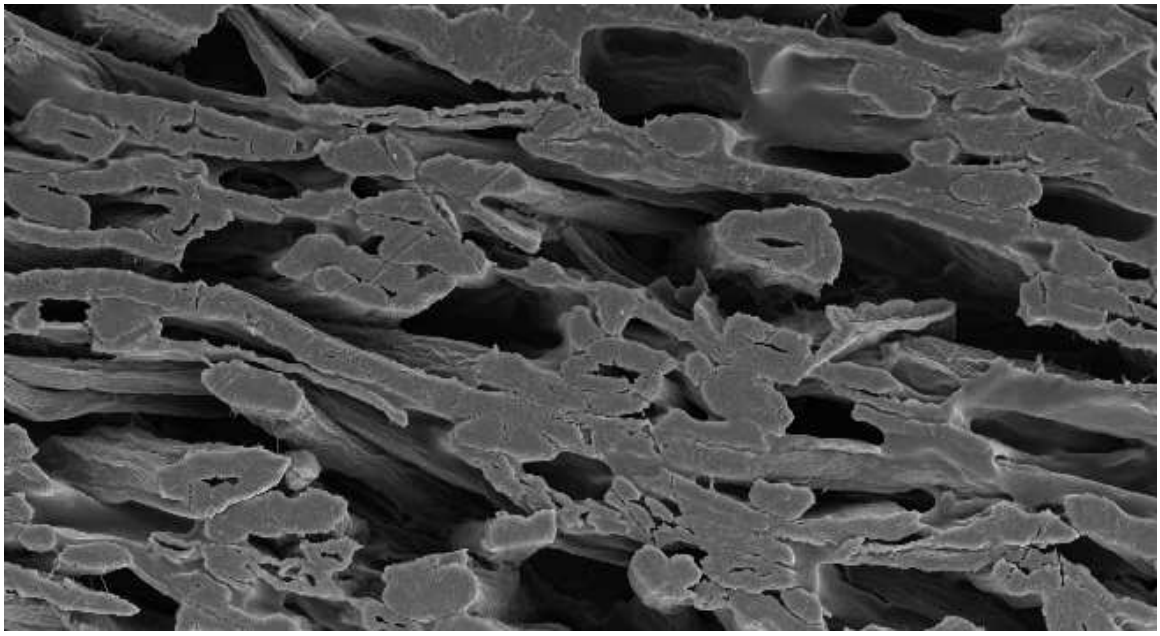


Illustration 23: Original micrograph before Binarization

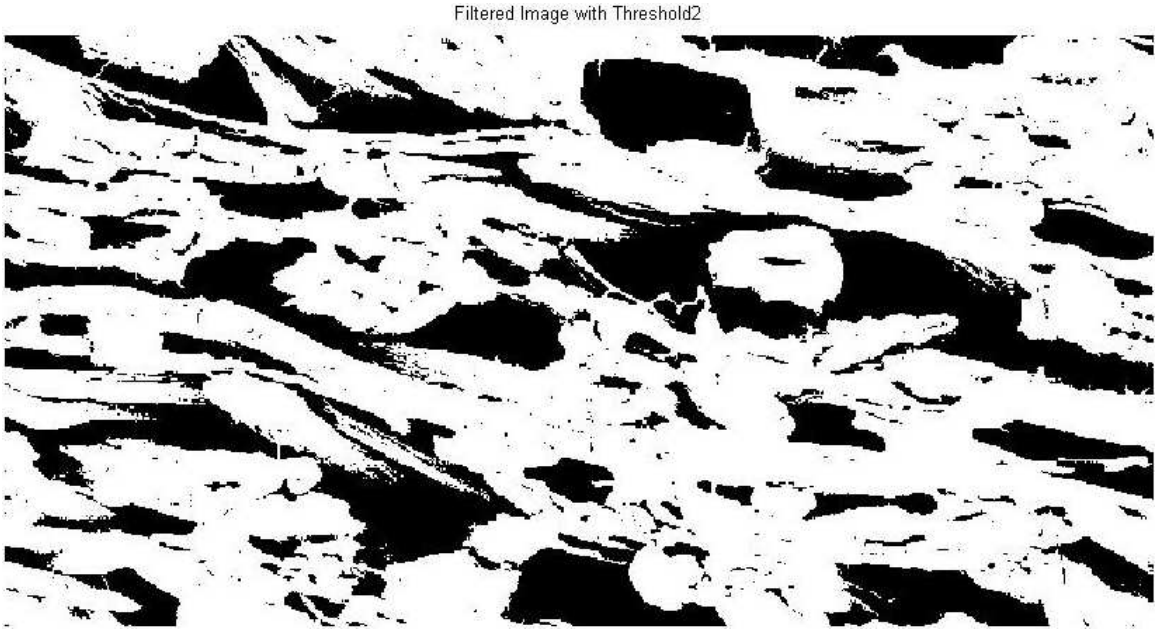


Illustration 24: Binarized sample

The lost of information concerning porosity is quite clear when studying this binarization.

Some other image analysis programs propose also a threshold range constituted of two values [T_{min}; T_{max}]. Here again, this is not sufficient in our case. It is simple to verify this when examining an intensities histogram of one particular micrograph. There are often at least three extrema that could fit the job as threshold value. That's why our goal here is to interpolate the intensities value in order to get a fitting cure on which we'll be able to extract extremum values.

For this purpose, one good thing to keep in mind is that we're not concerned by the accuracy of the interpolation, as long as we are close to the intensity values of concern. That's why we're not going to spend too much time on the interpolation. Using the built in functions *polyfit* and *polyval* in Matlab will be more than helpful for that purpose. Using a seventh degree polynomial to approach the intensities repartition is enough. From this stem point, it is then easy to determine the extrema using a differentiation method. We'll then determine at least 3 values for thresholding.

Determining three threshold values from a given enhanced intensities histogram has great advantages:

- We know that every intensity values under the lowest threshold called T1 will be part of a pore.
- We know that every intensity values above the highest threshold called Tmax will be material.
- We have then at least two different ranges on which we can cross check our pore determination using different techniques.
- In the case we have exactly three threshold values; we know we can still produce an intermediary binarized image using T2. Under this value, entities will be pores, above, they will be material. It can serve our cross check purpose as well.

Illustration on 3 level of Thresholding

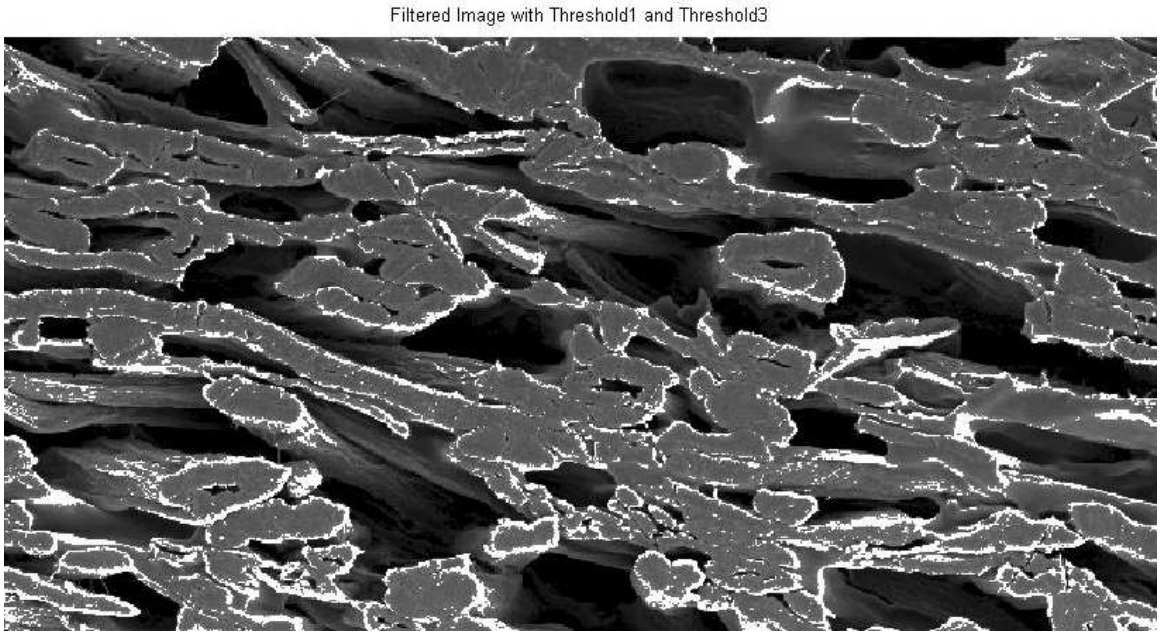


Illustration 25: Thresholding with extreme values

This approach has the advantage to preserve the overall geometry of the porous structure while optimizing it for an edge detection approach. Intensity values under a threshold T1 have arbitrarily been put to the value 0 while the values above a threshold T3 are being put to 1 (maximum normalized intensity – White).

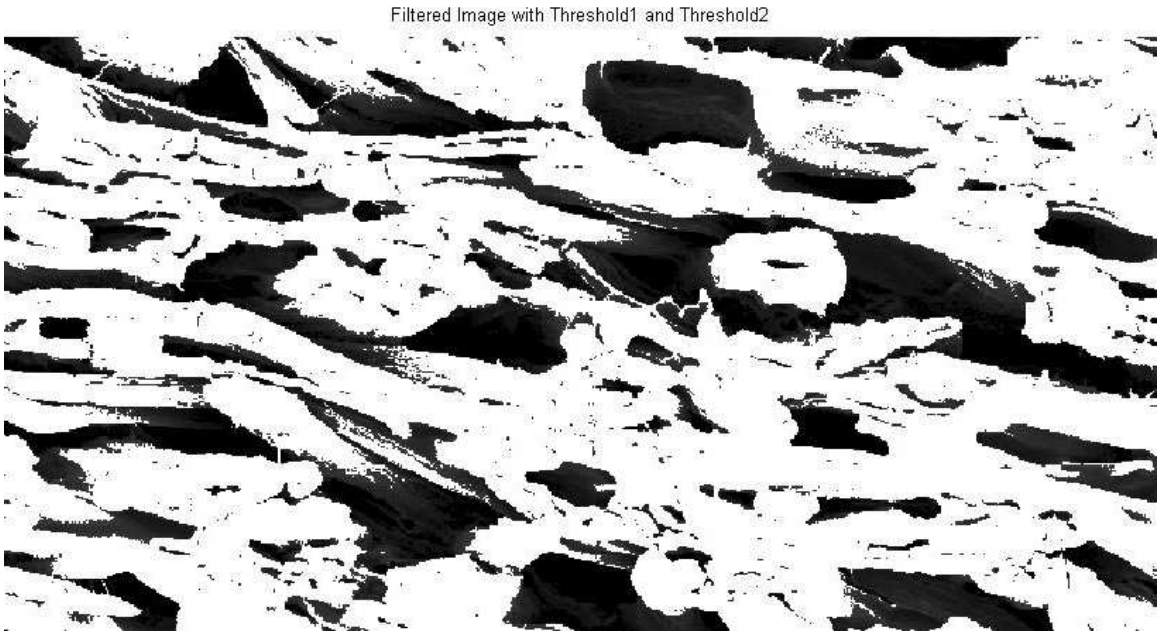


Illustration 26: Thresholding using lower values T1 and T2

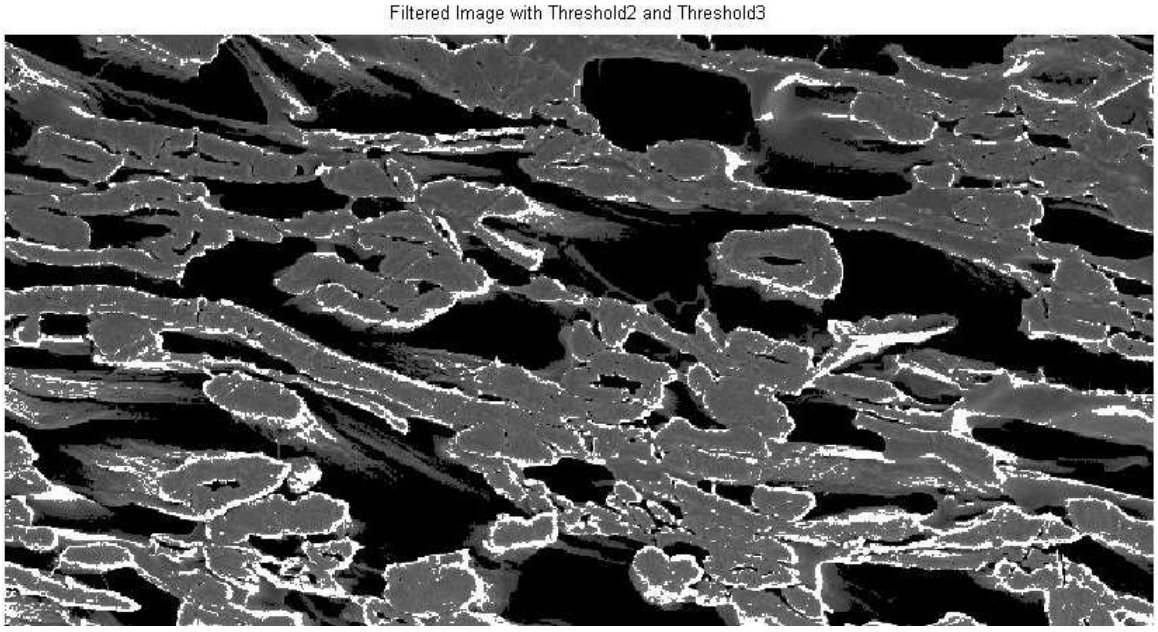


Illustration 27: Thresholding using upper values T2 and T3

The above operations have the interest to clearly define first the porous structure, then the fiber structure. One can manage both of approaches to optimize edge detection as well, hence optimizing the determination of porosity in such sample.

This approach is truly useful in enhancing the digital image at hand. Thresholding in this manner, we'll facilitate later on the job of edge detection for the purpose of pores determination.

Edge Detection Theory

The goal of edge detection is to check sharp changes in intensity gradients in order to determine shapes out of the overall image. In terms of Paper Science, it is then important to isolate features in a given sample taken from any imaging techniques. In a particular handsheet, the interest is thus to isolate the material part of the handsheets; whether it is fibers, fillers or any other chemicals; from the porous part. For the sake of simplicity, we'll talk only about fibers, however keeping in mind that the handsheet can be more complex and fibers can be coated with various elements.

General Principle

(*In the following, please refer to reference [18] for more information*)

Using edge detection has the advantage to minimize significantly the amount of data to be treated, while filtering any unnecessary information. There are essentially two different approaches to edge detection. The first one called search-base method detects edges by looking for maxima and minima in the first derivative of the image, usually local directional maxima of the gradient magnitude. The second one based on zero-crossing search zero-crossings in the second derivative of the image in order to find edges, usually the zero-crossings of the Laplacian or of a non-linear differential expression.

The first part of edge detection then requires the evaluation of derivatives of the image intensities. It is here a numerical problem which can involve various different type and different order of derivatives.

This process, as said before, often requires the regularization of the image intensities by filtering operations. It is of great importance in terms of well-posedness and numerical stability; two parameters that nobody can ignore when dealing with numerical operations.

(*In the following, please refer to reference [19] for more information*)

In 1979, Marr and Hildreth came up with some competitive criteria concerning edge detection that are important to underline in this thesis. They stated that intensity changes, which occur in a natural image over a wide range of scales, are detected separately at different scales. From that observation, they've determined that the second derivative of a Gaussian was the appropriate filter for this purpose. Their second statement was that intensity changes in images arise from surface discontinuities, or from reflectance or illumination boundaries; and these all have the property that they are spatially localized. Hence, intensity changes are detected by finding zero values of the Laplacian of a Gaussian

Application in Paper Web Analysis

Several edge detection methods have been implemented these last decades. This tool allows us to visualize boundaries between the two entities we wish to differentiate.
For this purpose, a built in function exists in Matlab, called *edge*. This function determines the edges in a gray scaled picture using several methods in parameter. Rather than detailing extensively the approach of the authors for such methods, we will review briefly them, and determine their interest in our case, which is determining the optimum choice to differentiate pores from fibers.

- ✓ The Sobel method finds edges using the Sobel approximation to the derivative. It returns edges at those points where the gradient of I is maximum.

- ✓ The Prewitt method finds edges using the Prewitt approximation to the derivative. It returns edges at those points where the gradient of I is maximum.
- ✓ The Roberts method finds edges using the Roberts approximation to the derivative. It returns edges at those points where the gradient of I is maximum.
- ✓ The Laplacian of Gaussian method (Marr and Hildreth) finds edges by looking for zero crossings after filtering I with a Laplacian of Gaussian filter.
- ✓ The zero-cross method finds edges by looking for zero crossings after filtering I with a filter you specify.
- ✓ The Canny method finds edges by looking for local maxima of the gradient of I. The gradient is calculated using the derivative of a Gaussian filter. The method uses two thresholds, to detect strong and weak edges, and includes the weak edges in the output only if they are connected to strong edges. This method is therefore less likely than the others to be fooled by noise, and more likely to detect true weak edges.

It appears after several analyses that our best choice is the Laplacian of a Gaussian method. Its first advantage is that it does not create artificially zero-crossing when the size of the filter is modified. In 2D, the Gaussian decomposes into the product of 1D Gaussians; hence it reduces consequently the number of computations involved.

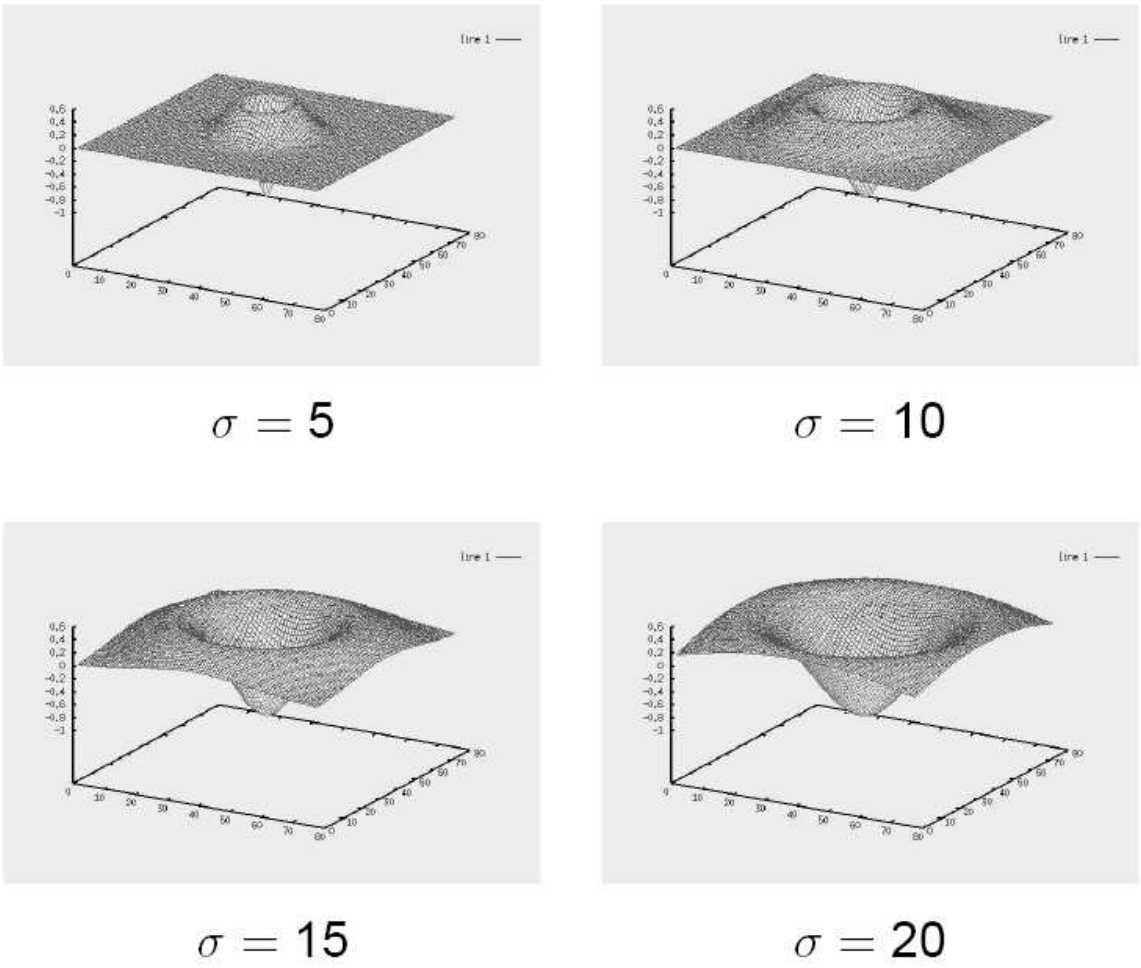


Illustration 28: Example of Laplacian of Gaussian filters

A First Approach Based on Thresholding

Due to specific needs concerning the model and characterization of starch reinforced paper with respect to both temperature and amount of starch, a specific program has been developed to obtain the following information:

- ✓ Overall porosity of the sample picture, i.e. ratio of pores to total surface.
- ✓ Key parameters of pores, i.e. either the radius if pores are circular in shape area or principal axes if pores are in elliptical shape with respect to thickness.
- ✓ Repartition of pores using key parameters on a histogram, applying a statistical criterion to get a smooth range on a graph (i.e. pores count and size)

- ✓ Rough estimate of connectivity between pores using a tolerance criterion on X and Y pixels.

The connectivity is here an important parameter since we want to determine the overall closure of the studied matrices with respect to the amount of starch in the handsheets. For this purpose, the program contains several subroutines as follows:

- ✓ The main program which runs every functions to analyze the desired micrograph.
- ✓ The function imAnalysis which loads the micrograph and runs a study of the overall contrast. This parameter induces an important bias in the study, so the procedure to filter the images has to be coherent for every study.
- ✓ The function bkPix which determines and saves in an array the amount of “black pixels”, corresponding to possible pores, scanning line per line the micrograph.
- ✓ The function connectivity which defines a criterion of connectivity with respect to fixed numbers of pixels in height and width of the micrograph. At this stage, a visual result is provided. The micrograph is transformed from a several level of grays to a color image where red pixels will stand for possible connectivity between pores.
- ✓ The function filtering has the purpose to determine which pixels are indeed parts of the connectivity criterion.
- ✓ The function porosity has somehow the same purpose of the function bkPix, only it’s putting together the black pixels that are indeed part of pores.
- ✓ The function poreAnalysis provides finally a specific count of pixels per pore, thus summarizing the overall porosity after filtering the image, which can be compared to a rough estimation of porosity performed with the function imAnalysis.

Steps of Computation

Image Analysis process

As a first step, the program reads the desired micrograph in order to apply the different image processing tools. Each micrograph is in ".TIFF" format which keeps the desired precision for study. They're all based on a gray scale representation ranging from 0 to 255, which is to say from black to white pixels. The amount of pixels per specific value of gray determines the intensity of each image.

A first approach is to determine edges in order to differentiate fibers and fines from pores, filled or not with starch. This method uses different method of gradients known as Canny, Sobel, Prewitt or Roberts Method (The MathWorks 2006). The best method was to use a double Gaussian Filter along X and Y-axis. However, a further study shows that these methods are not quite adapted to the problem since we lose the information of what is a pore or what is a fiber.

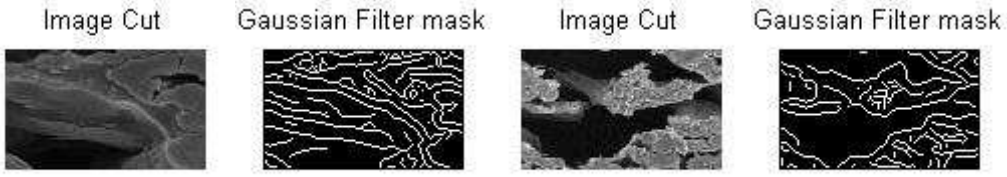


Illustration 29: Early study of edge detection (not conclusive)

A second approach was then to determine a threshold which would be applied as a criterion to determine what can be considered as part of a pore or as part of fiber matrix. In order to achieve this goal, a study of contrast was performed. Several functions are proposed in Matlab Toolboxes for image analysis, however, here again, we wanted to keep a certain control of the procedure in order to stay coherent for any further study.

For this purpose, the function imAnalysis runs three different type of equalization based on the following methods:

A first approach is based on a “classical” equalization which smoothes the histogram of intensities over the entire range of gray levels. This method is of course less well adapted to our problem since it’s modifying linearly the information contained in a micrograph which yields a lost of information.

A second approach makes use of adaptative histogram equalization with two different distributions: Exponential or Rayleigh Distribution.

Illustration on a specific example:

The following is the case of a sample containing 20% of starch pressed at 300°C.

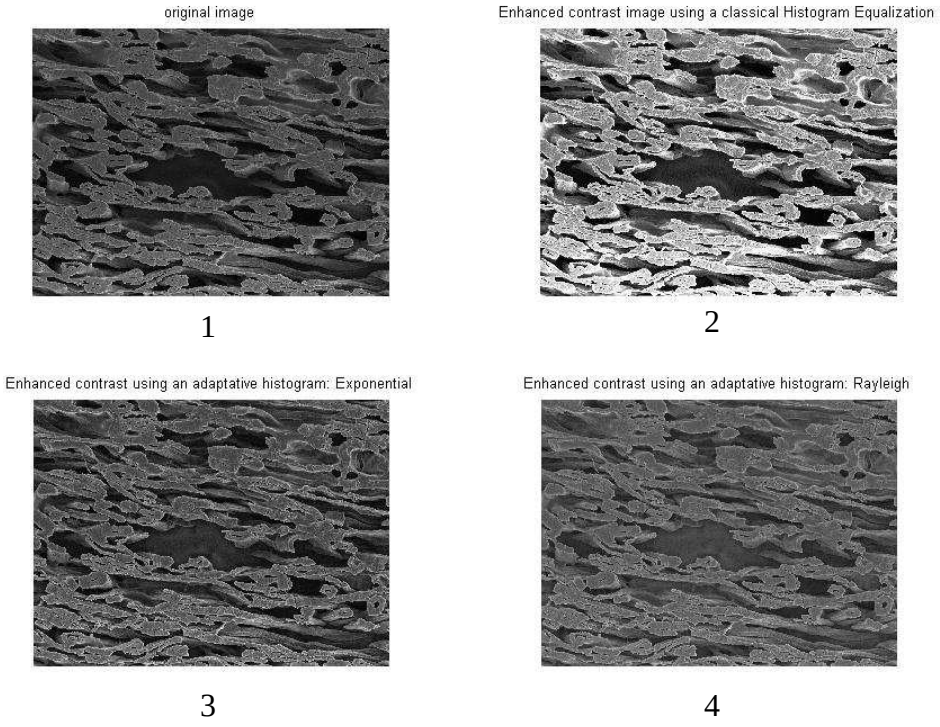


Illustration 30: Early study of contrast enhancement

#1: original micrograph.

#2: Micrograph enhanced using standard equalization.

#3: Micrograph enhanced using adaptative exponential distribution.

#4/ Micrograph enhanced using adaptative Rayleigh distribution.

The principle of Contrast-Limited Adaptive Histogram Equalization operates on small regions in the image, called tiles, rather than the entire image. Each tile's contrast is enhanced, so that the histogram of the output region approximately matches the histogram specified by the 'Distribution' parameter. The neighboring tiles are then combined using bilinear interpolation to eliminate artificially induced boundaries. The contrast, especially in homogeneous areas, can be limited to avoid amplifying any noise that might be present in the image. These are built in function from Matlab Software ©.

The results can be shown on histograms to observe the effective procedure of each contrast enhancement:

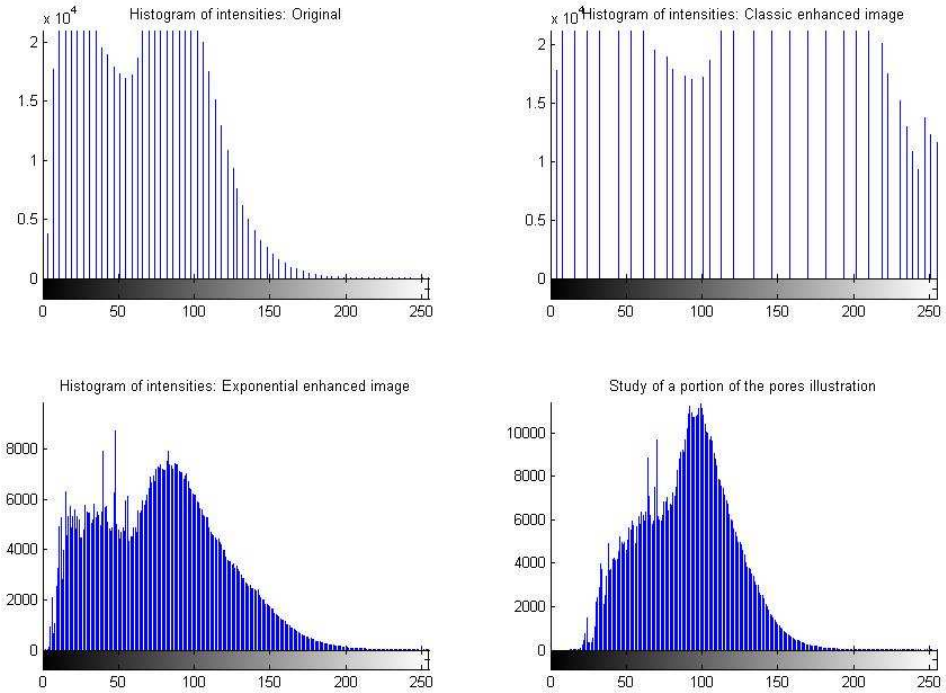


Illustration 31: Histogram of intensities for any sample

As observed on the top right illustration, the original enhancement is useless for the study which consists again to determine as precisely as possible: what range of gray levels defines pores in the matrix and what range defines then fibers and fines.
The choice between Exponential and Rayleigh distribution (Bottom two illustrations) is determined by the apparent repartition of gray levels over the global range [0 255].

Thus, the Exponential distribution determines the best choice since the lower values which corresponds to the darkest level of gray, hence the pores, are preserved while the higher range is smoothed. While Rayleigh distribution works more as a Student stochastic distribution and thus sharpen the higher values as somehow truncating the lower range of values.

Hence, the threshold is determined based on this contrast enhancement techniques as 2/3 of the gray level value which itself corresponds to the maximum range of intensities.

Example: studying the Histogram of intensities with Exponential enhancement, we have an apparent range [0 141] where the intensities are most likely to be found. Now taking 2/3 of this maximum value, we end up with an apparent range for pores study of [0 94]. Thus, the threshold for this micrograph is 94, under which values of gray level are considered part of a pore.

Further on this contrast analysis, one can be concerned about the “tiles” analysis provided by the adaptative method. This parameter can be investigated to determine an optimum value. For the study of porosity, this is a crucial parameter since it will determine later on our criterion of connectivity. Hence, optimum tiles have been found for our case to be 6x10 pixels. This parameter applied to the contrast-limited equalization gives the following results:

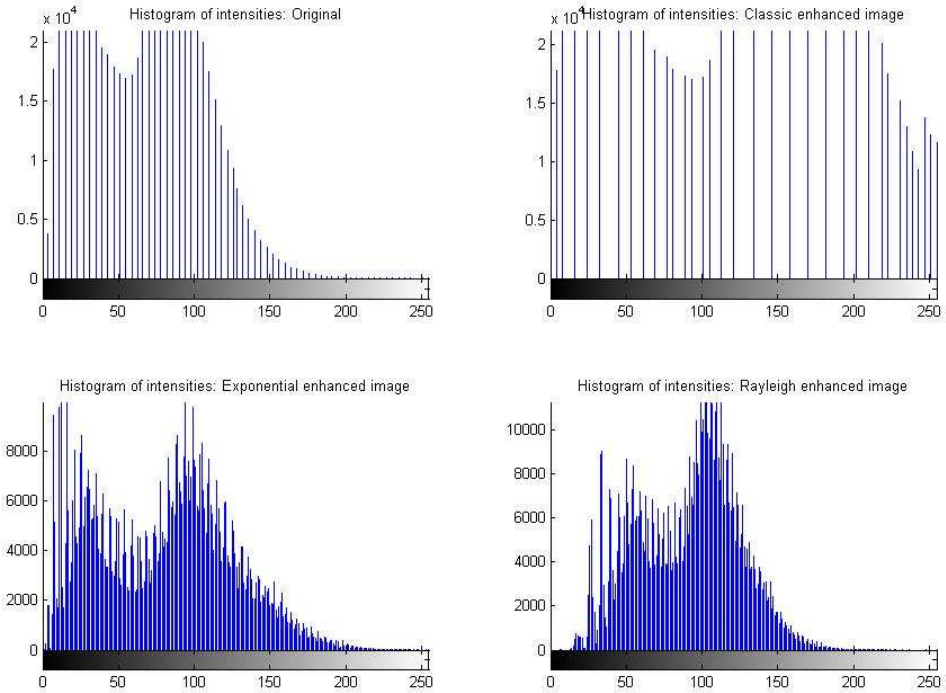


Illustration 32: Adaptative equalization on intensities

Although they might appear a bit messy, the determination of the threshold based on these equalizations appears to be more precise.

The following illustration of fibers for the micrographs called 23_SEM.TIF which corresponds to our current example (i.e. an impulse dried handsheet at 300^{o}C, containing a relative amount of starch of 20%).

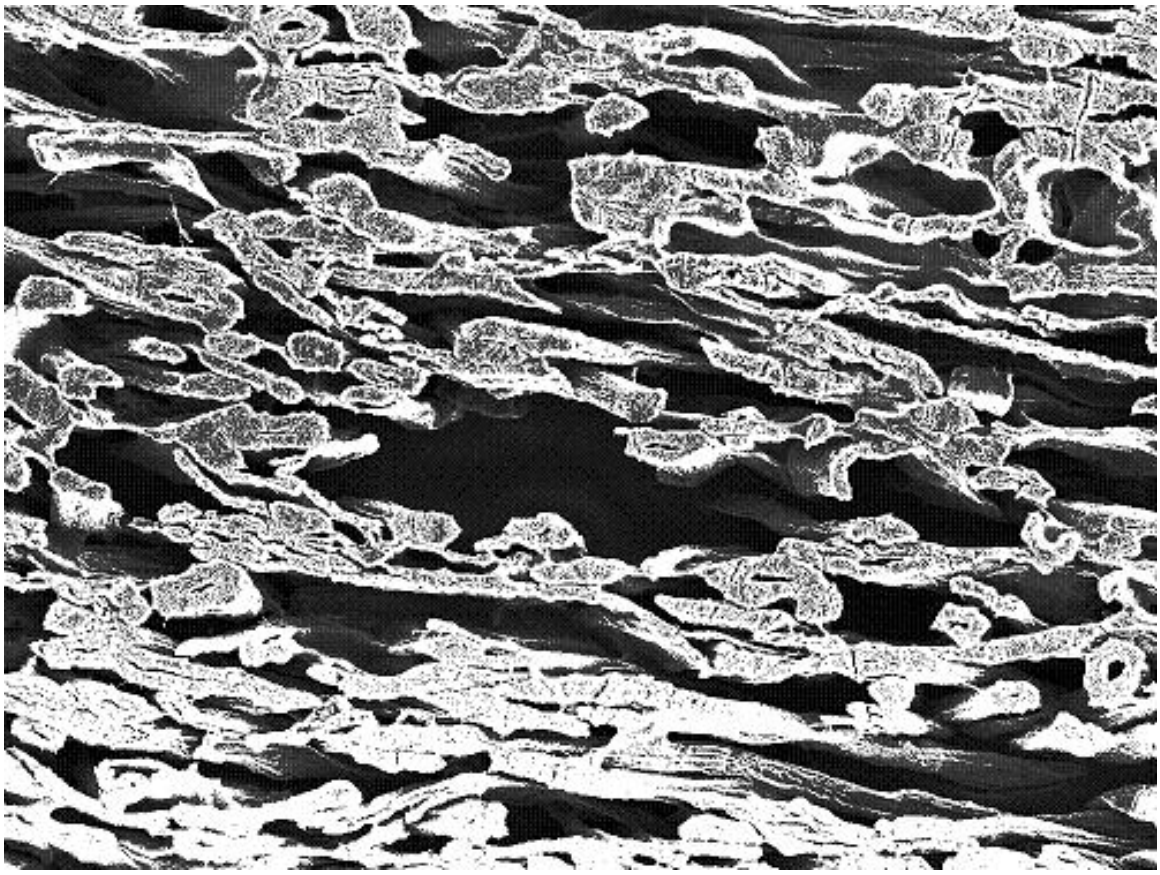


Illustration 33: Binarized sample

Compared to the original:

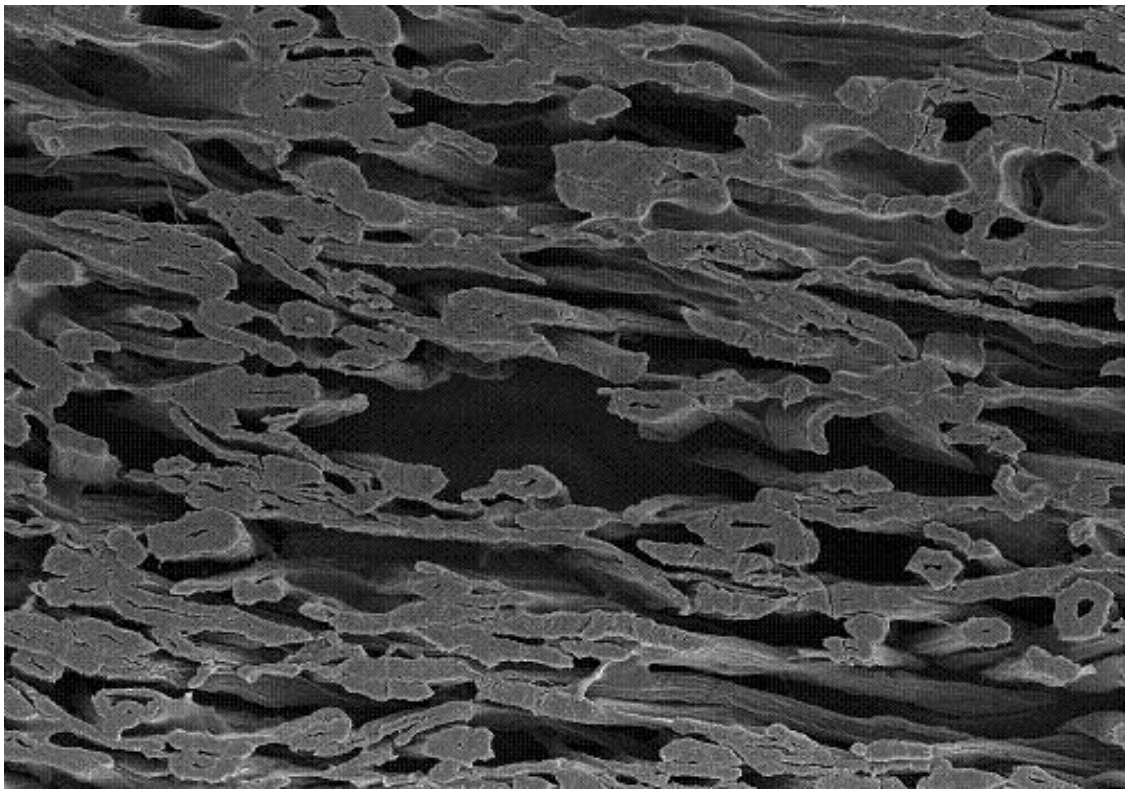


Illustration 34: Original - 20% starch imbued handsheet, impulse dried at 300°C

We now clearly see the structure of pores versus fibers without losing any detail of the global micrograph. Although the apparent lack of precision to illustrate fibers in the enhance version might be confusing, one has to keep in mind that our aim is to determine apparent porosity, in other words we have to keep in mind that we want to keep the fibers we see on top or “surface fibers” apart from pores which have clearly here a depth component, which at this stage of the study we can’t characterize.

This is where the criterion of connectivity takes all its sense. We have to finish the reconstitution of the fibers while keeping pores as they appear. For this purpose, a second function is used: bkPix which is detailed in the next step.

Constitution of an Array of black pixels

This part plays an essential role in the determination of pores and their location. The principle here is to scan the image modified using contrast adaptative equalization line by line and count precisely every pixels being part of a pore as defined by the threshold. At the same time, the line number and beginning plus ending columns are recorded.

Here is a short example of the result:

# of black pixels	40	21	2	30	2	4	23
column beginning #	1	45	75	123	174	188	214
column ending #	40	65	76	152	175	191	236
line #	1	1	1	1	1	1	1

Figure 11: Black pixel count for the determination of pores

The first line of the array gives the total number of black pixels encountered as part of a pore.

The second line gives the number of the beginning column.

The third line gives the number of the ending column.

The fourth line gives finally the number of the line in the micrograph.

This information is then saved in the Workspace with all the other essential variables in order to be accessible by any function making use of these variables. The importance of this procedure is to highlight the fact that now we need to apply somehow a connectivity criterion. Look for example at line 1, column 75 to 76: we encounter only two pixels here, which might not be part of a pore, or differently put, they're not relevant in pores determination since we cannot expect the precision to be this low in the micrograph.

That’s where the connectivity criterion comes: the next step is to determine with respect to this criterion which pixels might be either part of connectivity between two different pores, or as seen on the micrograph 23_SEM.TIF previously part of fibers pixels which are not yet validated.

Example:

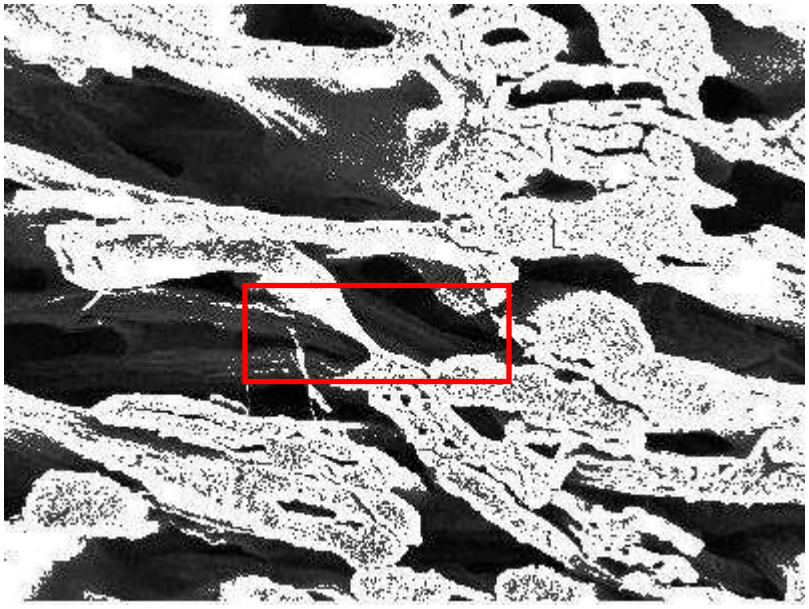


Illustration 35: Illustration on connectivity issue

Focusing on a small part of sample 23_SEM, we clearly see that the region in the red rectangular has to appear all in white pixels, as part of a fiber. That’s where the connectivity criterion will help to determine if isolated pixels are relevant to pores count or simply part of fibers.

Image analysis in color for connectivity check

Here again, an important step is to visualize what modifications are done to the micrograph in an effort to keep coherent along the analysis.

That's why a first modification is to now to transform the previous micrograph from gray level intensities representation to a fully colored image, typically in RGB representation (standing for Red-Green-Blue). This modification offers the opportunity to increase the precision of analysis, allowing us to work actually on a "three level" matrix.

To be more exact:

- Previous micrographs were in TIF format using only gray levels representation. This is to say, each pixel is assigned a value between 0 and 255. O stands for black color while 255 stands for white. So finally, we're dealing with a matrix of "height x width" pixels in a range [0 255]
- Further micrographs are using RGB representation, so we're now talking of 3 matrices instead of one, ranging again from 0 to 255 which gives for a pixel for example: Black = [0 0 0], White = [255 255 255], Red = [255 0 0] …

The choice is now to assign a specific color value to any pixels which are possibly either part of a fiber, either connectivity between two different pores. An arbitrary choice has been to show them in red for a preliminary study, in an effort to distinguish them from the rest of the image. We're talking again of an intermediate modification in order to keep coherent with any analysis and then be able to talk about repeatability in pores determination. Red pixels are determined with respect to the connectivity criterion, which is made easily using the array previously created: we only have to highlight any pixels which are under the tolerance in Y direction (or width of the image), which is any total of consecutive black pixels inferior to 10. (Recall: connectivity criterion is 6x10).

Note: Keep in mind that under Matlab conventions, X-direction stands for the height of the image while Y-direction stands for the width. These are not conventional axis choice!!

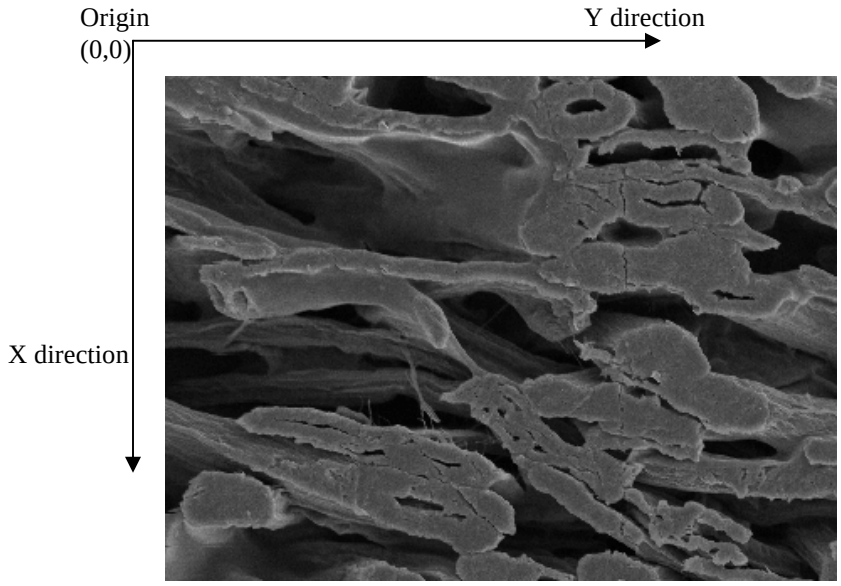


Illustration of Connectivity check

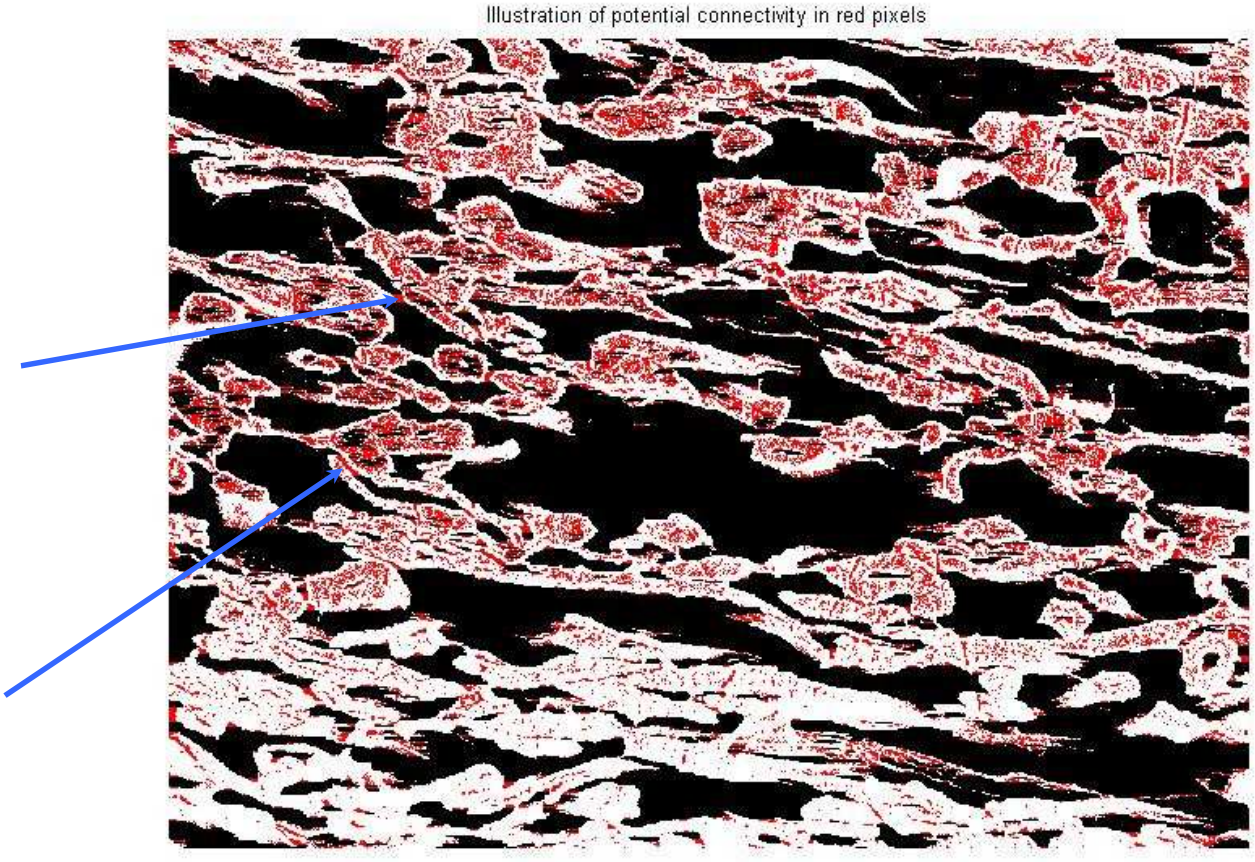


Illustration 36: Connectivity designated by the blue arrows

We now see that without touching the overall repartition and shape of pores, a lot of work has to be done to somehow recreate what are fibers and precisely determine what really connectivity is between pores, as shown by the arrows.

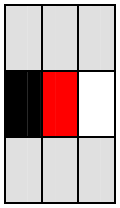
Note: Particular attention has to be placed on red pixels that might appear as part of a pore but do not fit any of the above cases. From now on, it's not treated as it should, so the pores count contains a small error that is repeated for any analysis. It can be considered as part of the information lost while working on contrast of the micrograph.

Filtering the connectivity image

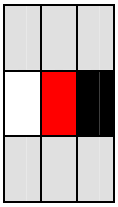
The issue now is to treat each red pixel one by one and determine with respect to some criteria what do they belong to.

Here is a detail of what has been arbitrarily decided to analyze the new color image:

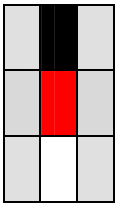
i- Alternate pixels: *gray pixels in the following example can be anything.*

If this situation occurs: 

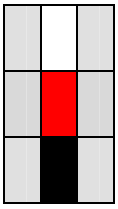
→ Then the red pixel is considered as part of a pore, i.e. black.

If this situation occurs: 

→ Then the red pixel is considered part of a fiber, i.e. white.

If this situation occurs: 

→ Then the red pixel is considered as part of a pore, i.e. black.

If this situation occurs: 

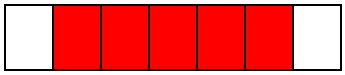
→ Then the red pixel is considered part of a fiber, i.e. white.

This procedure will be repeated at the end to make sure any single pixels are considered as part of a pore or a fiber.

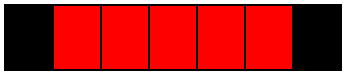
ii- Series of 5 pixels.

Now bigger features are to be considered. Especially, in case they create more parasite features after treatment, we want them to appear before a finer filtering.

At this stage, a simple case is considered: only both ends of the series are considered.



→Then the entire set is considered as white pixels.

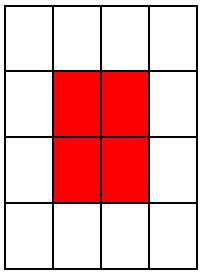


→Then the entire set is considered as black pixels.

Since the image is scanned line by line, for this last feature, there is no need to scan for series of 5 red pixels in a column. This would lead to a loss of information from the original micrograph.

iii- Set of 2x2 red pixels: only for fibers.

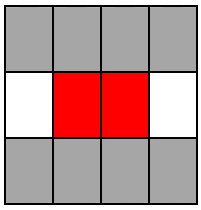
In order to recover more pixels from fibers which may appear as part of a hole because of the choice of the threshold, we scan now the modified image for square shapes, which will increase the recovering process for fibers.



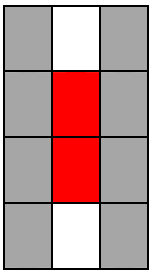
→ Then the set is considered as part of a fiber.

After several tries, it appears that at this stage, the modified image can be scanned now for smaller features without losing any more information at all. Choice of either recovering pores pixels or fibers pixels will be compensated each other in order to stay in an acceptable range of error.

iv- Case of pair of red pixels.



→ Then the set is part of a fiber, whatever lies above or underneath that set.



→ Then the set is part of a fiber.

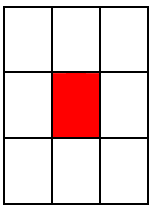
The same procedure is applied in case pixels at both ends are black, then the pair will be considered as part of a pore and modified consequently.

v- Set of three red pixels.

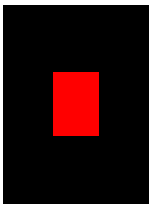
The exact same procedure as above is applied.

vi- Single red pixel.

A final screening of the modified micrograph is made considering single red pixel that is obviously either part of a pore or a fiber.



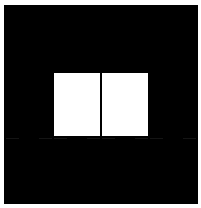
→ Red pixel part of a fiber.



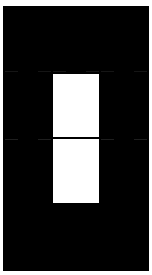
→ Red pixel part of a pore.

vii – Non relevant pixels.

A final screening has to be performed to check about black and white pixels which are not relevant for the study:

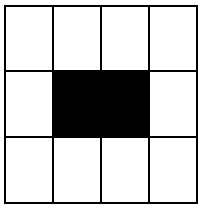


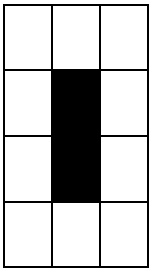
Or



Then the pair of white pixels is modified to be part of the pore.

Or





Then the pair of black pixels is modified to be part of a fiber.

Finally, the same principle is applied to get rid of irrelevant single black or white pixels.

Illustration of filtering:

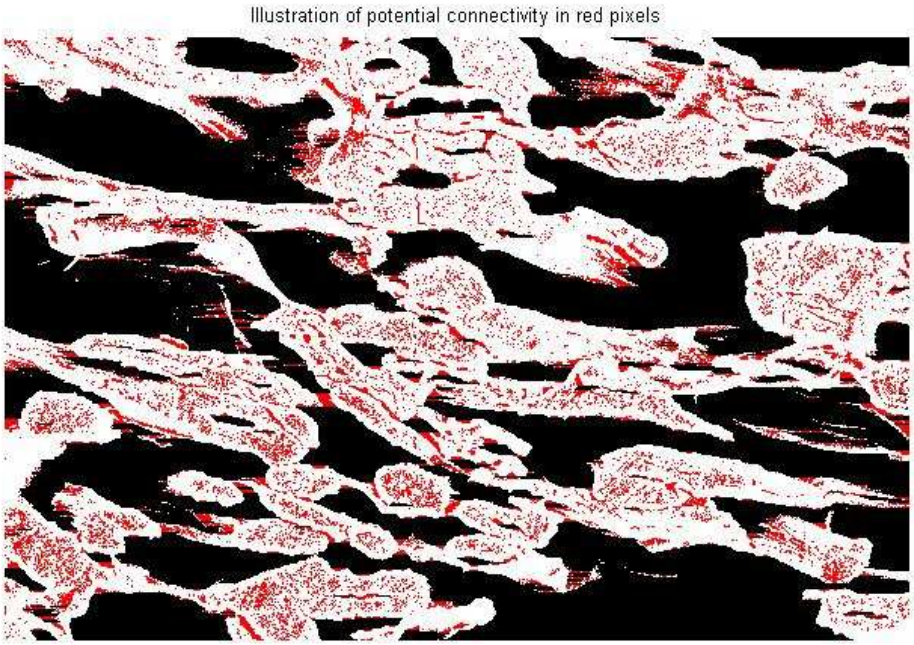


Illustration 37: Image with potential connectivity

Here is illustrated again the possible connectivity in red pixels on the micrograph labeled 23_SEM.TIF. For convenience, only a portion of the entire micrograph is displayed: 400x600 instead of 750x1000

After filtering:

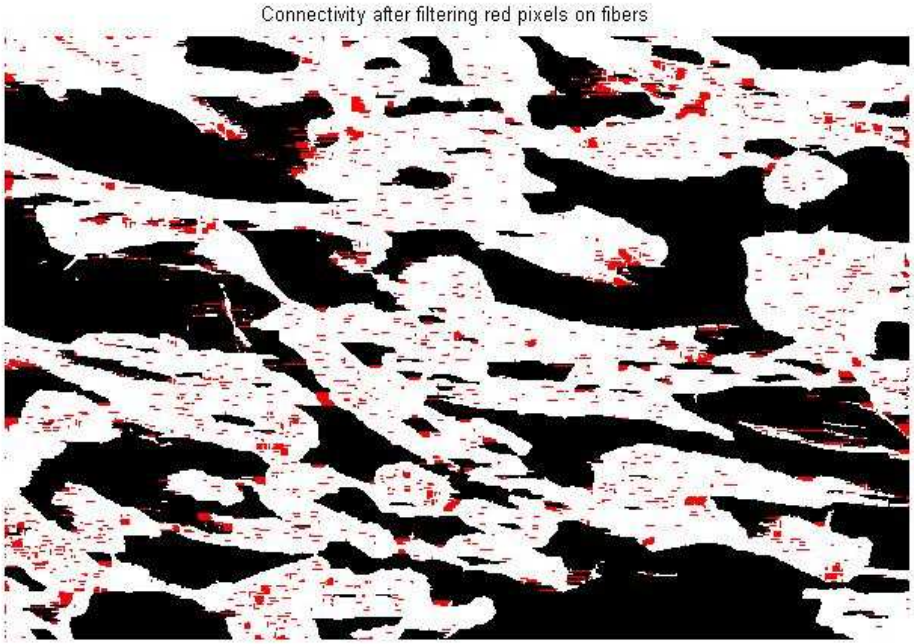


Illustration 38: Filtered image with a clearer view on potential connectivity

Even though some irrelevant red pixels are still not treated, the overall image is now acceptable for porous analysis and keeps essential connectivity between pores. From the original, we can observe that we have reconstructed the fibers fairly precisely in order to increase the precision on global porosity determination, which will be compared to the first rough estimation processed by the function imAnalysis at the beginning.

Note: Obviously at this point, an optimization is possible concerning the way the micrograph is filtered in order to improve the reconstruction of the micrograph. However, as said before, the reconstruction is acceptable in terms of errors.

Preliminary pore analysis

This intermediate step before the precise determination of pores in the micrograph is intended to lower the computing time of the overall process.

This step consists in a similar manner of the same process descried for the function bkPix. The goal here is to recollect every black pixel that is now definitely part of a pore. Since we're working now on a color image, the scanning has to skip both red and white pixels, i.e. [255 0 0] or [255 255 255] in terms of color code. As we can see, the simplest way to do so here is simply to check for the first value since both red and white pixels should be 255 while the value for black pixel is 0.

Each useful value is then recorded in an array called porous1 which has 4 lines standing respectively for:

- ✓ The total number of black pixel encountered between two non-black pixels in a line.
- ✓ The number of the column where the counting begins.
- ✓ The number of the column where the counting ends.
- ✓ The number of the line scanned.

Illustration on the micrograph 23_SEM.TIF:

35	18	14	23	131	38	24	14	21
122	270	342	403	470	122	265	343	405
156	287	355	425	600	159	288	356	425
1	1	1	1	1	2	2	2	2

Figure 12: Count of black pixel for pores determination

As we see for this micrograph, the first pore encountered starts at (1,122) and its first line counts 35 black pixels between column 122 and column 156.

Note: This can easily be verified on Matlab typing the command:
imColor(1,122,:) which displays the three RGB values of the pixel (1,122) under the convention described in the program. If it's indeed a black pixel, it should display [0 0 0]. Some precautions have been taken for this purpose in this function.
Here again, the workspace is saved for the next step.

Color Convention for the following functions:

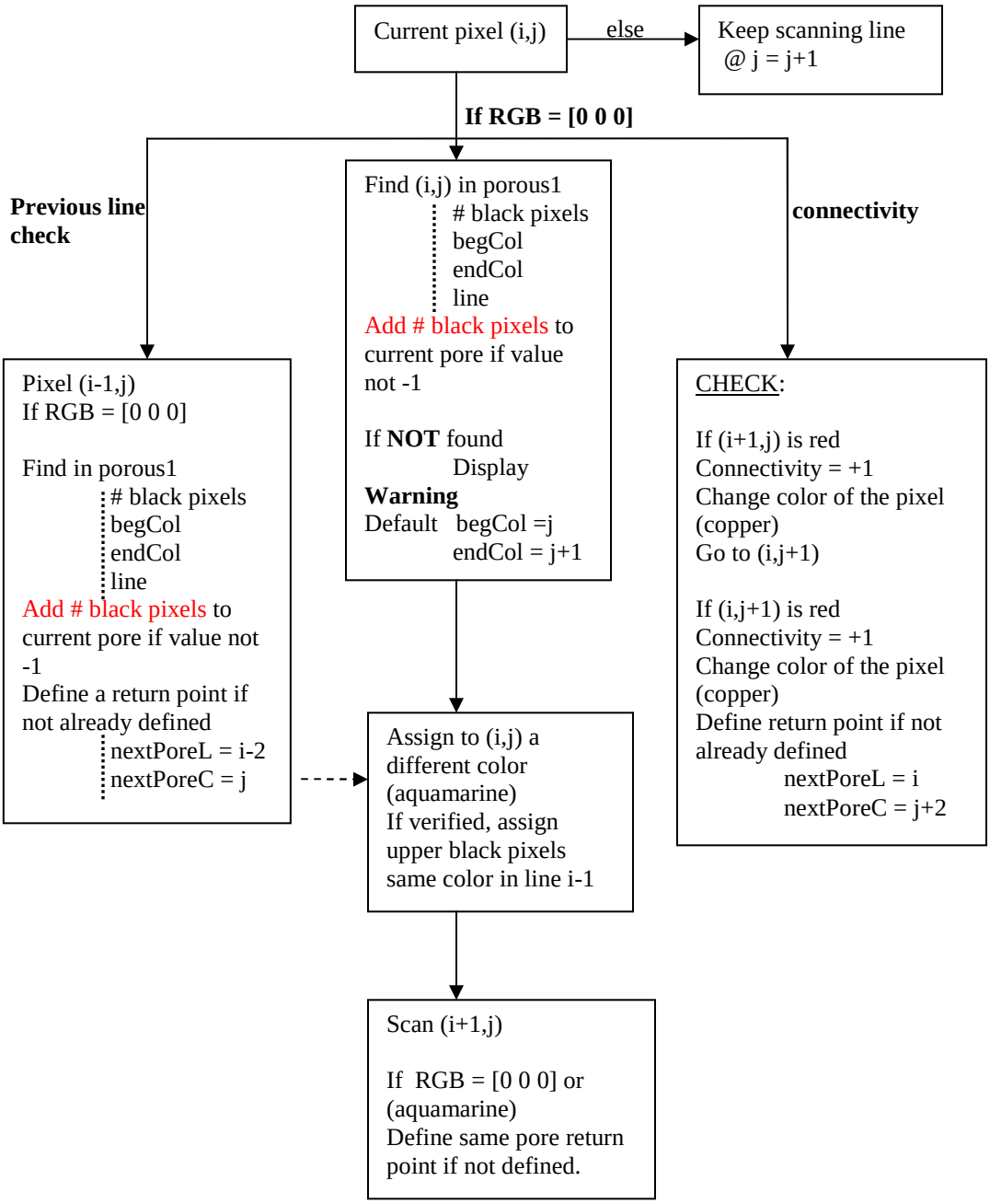
Color	**Feature**	**RGB code**
Black	Pores	[0 0 0]
White	Fibers	[1 1 1]
Red	Possible connectivity	[1 0 0]
Aquamarine	Filtered pores	[0.49 1 0.83]
Copper	Final connectivity	[1 0.62 0.40]

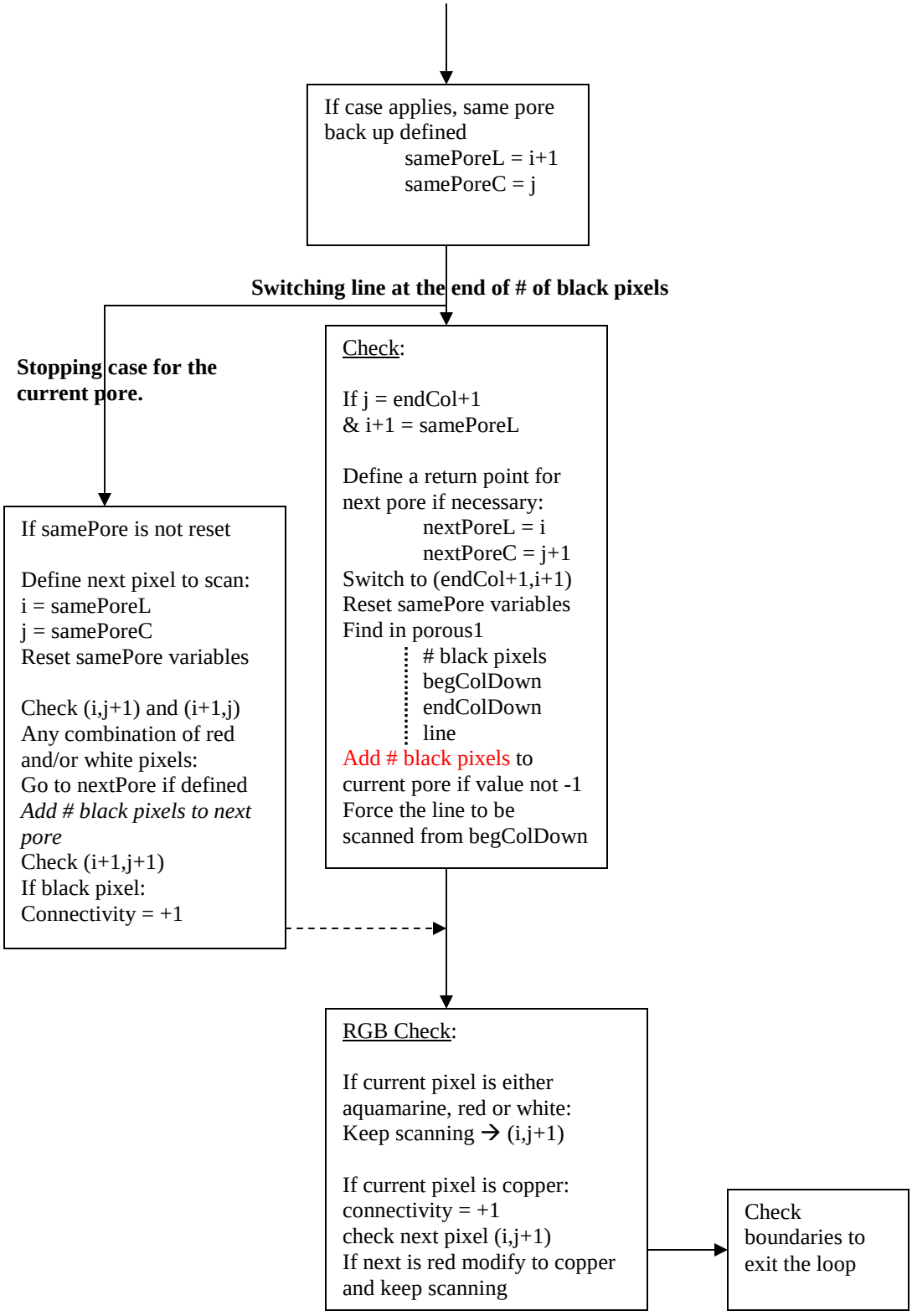
Figure 13: Color convention

Function porosity for the determination of pores in the micrograph

This function is the key stone of the program: it will determine precisely each pore of the micrograph one by one and specify its total area while checking for possible connectivity along the screening.

Functionality:




If case applies, same pore back up defined
samePoreL = i+1
samePoreC = j
Switching line at the end of # of black pixels
Stopping case for the current pore.
Check:
If j = endCol+1
& i+1 = samePoreL
Define a return point for next pore if necessary:
nextPoreL = i
nextPoreC = j+1
Switch to (endCol+1,i+1)
Reset samePore variables
Find in porous1
black pixels
begColDown
endColDown
line
Add # black pixels to current pore if value not -1
Force the line to be scanned from begColDown
If samePore is not reset
Define next pixel to scan:
i = samePoreL
j = samePoreC
Reset samePore variables
Check (i,j+1) and (i+1,j)
Any combination of red and/or white pixels:
Go to nextPore if defined
Add # black pixels to next pore
Check (i+1,j+1)
If black pixel:
Connectivity = +1
RGB Check:
If current pixel is either aquamarine, red or white:
Keep scanning → (i,j+1)
If current pixel is copper:
connectivity = +1
check next pixel (i,j+1)
If next is red modify to copper and keep scanning
Check boundaries to exit the loop

The function porosity finally provides an illustration of how pixels have been analyzed as illustrated:

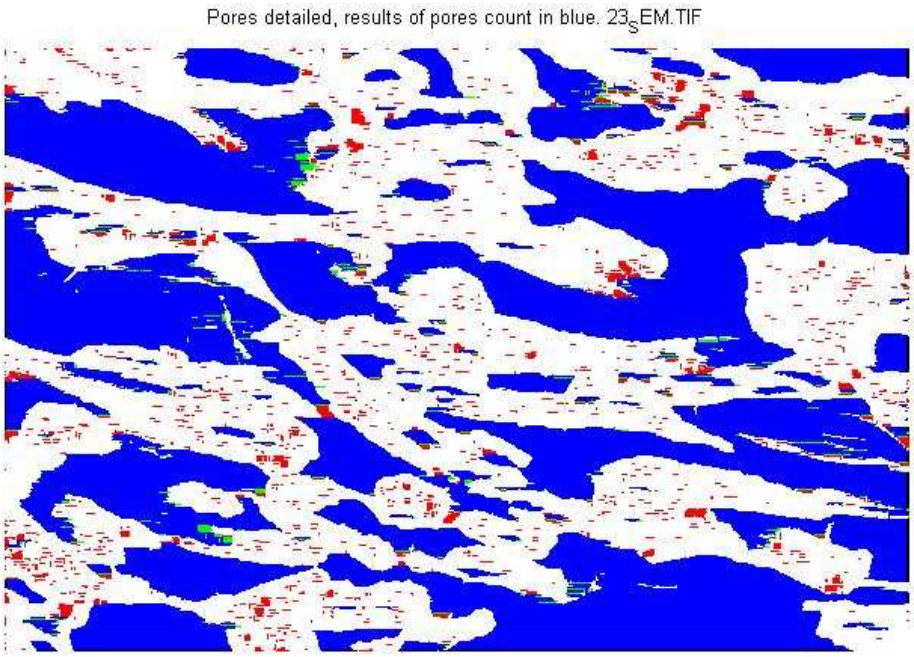


Illustration 39: Detail of pore determination (blue = pores; red = possible connectivity)

Although optimization has still to be considered about the connectivity criterion, pores are all determined (Example using previous syntax for colors).

As described in the algorithm, the principle of this function is to make use of the previous array computed in porous1 to determine exact amount of black pixels and position along lines and columns which are scanned from origin (top left corner) to last pixel (Xpix, Ypix).

Next function is the main function which runs every subroutine and runs statistical analysis of the pores repartition.

Main function

The purpose of this function is mainly to provide an automated way to analyze results about porosity.

The principle is to access a word or text file which contains respectively the *FileNames.TIF*, the resolution of one pixel, the height and width of the micrograph. This file is then read and an array is created containing all these information that a user can choose:

```
>> main
List of Micrographs:
1       1_SEM_001

2       8_SEM

3       19_SEM

4       23_SEM

5       73_SEM_001

6       73_SEM_002

7       73_SEM_003
```

In case the user wants to choose a more precise analyze, entering either 0 or any other characters that don't fit the list of numbers in the list, a prompt provides then the possibility to choose the micrograph and enter all parameters as illustrated:

```
Enter the number of the micrograph to study: 0
Enter the micrograph file_name.TIF to study: 23_SEM.TIF
Enter the resolution of one pixel: 0.314
```

Enter the height of the image you want to study: 250

Enter the width of the image you want to study: 200

A summary of the choices is then displayed:

ans =

Your choice is the micrograph: 23_SEM.TIF

with a resolution of 0.314 µm x 0.314 µm per pixel

Size of the micrograph studied is 250 x 200

To summarize the analysis, a figure is provided for comparison between the original micrograph and the scanned image:

Illustration 40: Final micrograph after pore analysis

Example taken from micrograph 23_SEM.TIF, size: 400x600

Pore determination can then be illustrated in different ways:

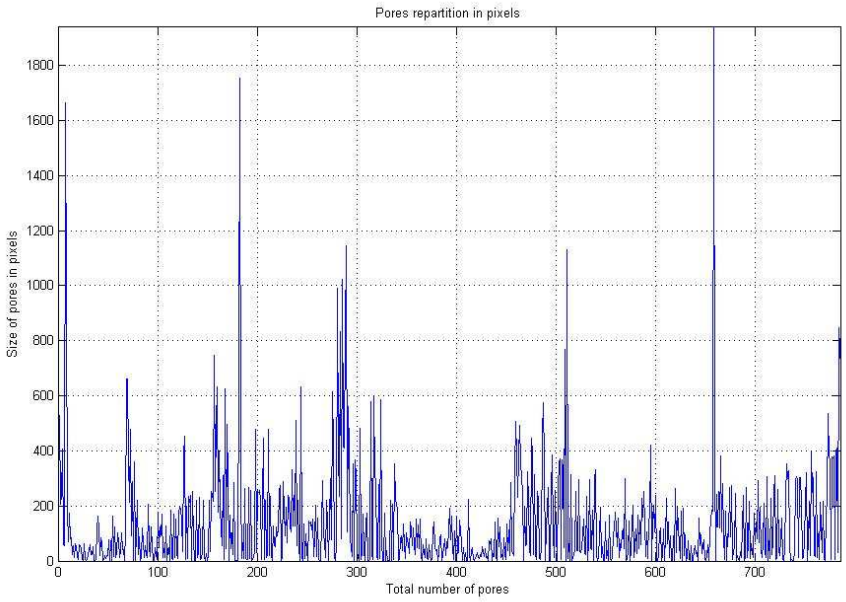


Illustration 41: Pores-size distribution as encountered in the micrograph

Here is a rough repartition of pores with respect to the analysis, results provided in total pixels.

A more convenient analysis is to run a statistical analysis which will give the mean and the standard deviation on pre-defined categories.

Categories are defined as follow:

- ✓ Determine the biggest pore.
- ✓ Define ten categories; each one will contain pores which have a total area less than half of the maximum from previous category, except for the too small pores area.
- ✓ Exact amount of pores per category is indicated.

Illustration using micrograph 23_SEM.TIF, size 400x600

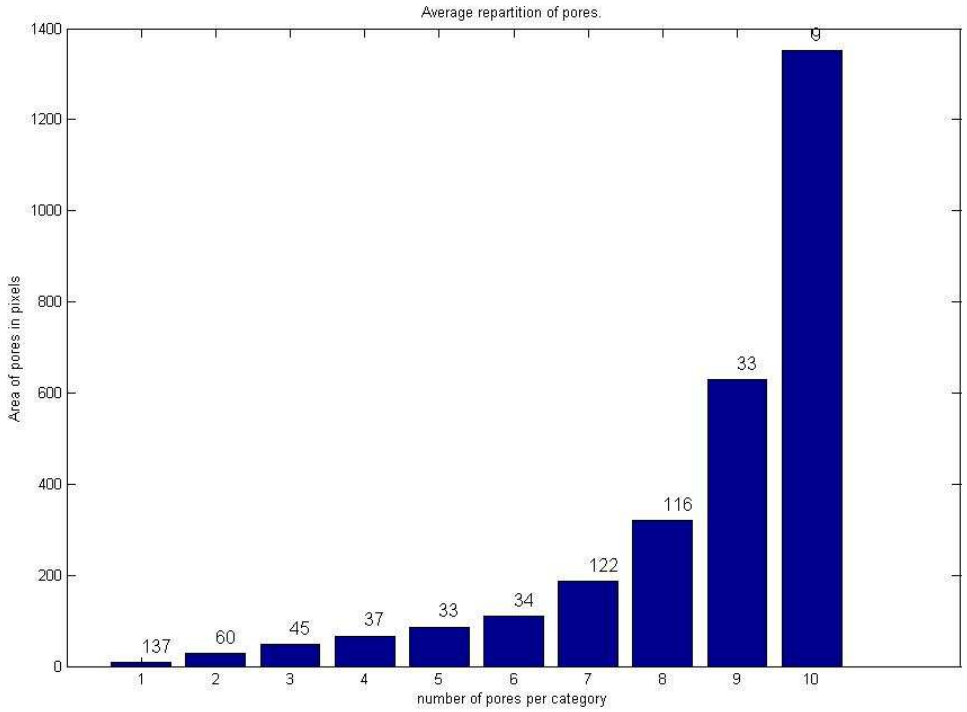


Illustration 42: Ordered pore-size distribution

Category 1: [0 1%] → 137 pores

Category 2: [1% 2%] → 60 pores

Category 3: [2% 3%] → 45 pores

Category 4: [3% 4%] → 37 pores

Category 5: [4% 5%] → 33 pores

Category 6: [5% 6.25%] → 34 pores

Category 7: [6.25% 12.5%] → 122 pores

Category 8: [12.5% 25%] → 116 pores

Category 9: [25% 50%] → 33 pores

Category 10: [50% 100%] → 9 pores (Macropores)

Percentages are taken with respect to the maximum value of total area. In other words, the biggest pore in the micrograph serves as the reference to define categories 1 to 9. This kind of illustration gives the opportunity to compare the global repartition of pores and their mean size per categories. Here, we can see that the first majority of pores are less than about 20 to 30 µm², the next big category of pores are in between 200 to 300 µm², while there is a total of 9 macropores with a size of about 1300 µm². These results refer to the case of a 20% relative content of starch in a handsheet impulse dried at 300°C.

Another way to compare results and mean repartition is to sort the array containing pores sizes and plot the curve along with mean reparation:

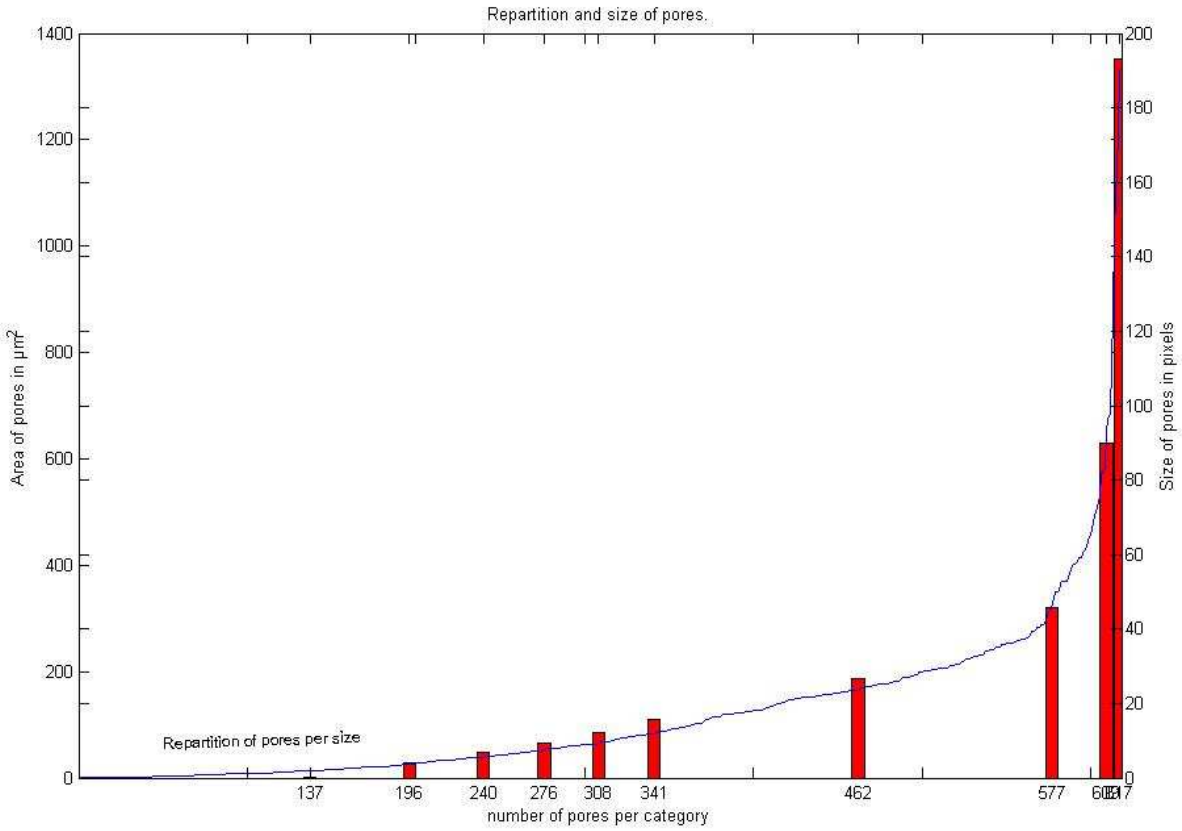


Illustration 43: pore-size distribution per pore count on a given micrograph

Here the results are provide in µm² concerning the mean repartition (red bars) and in total area pixels (blue curve). The x-axis provides the total number of pores encountered from the smallest pores to the biggest. Hence, there are about 462 pores with a size less than 200 µm² and the biggest pores are about 1300 µm².

Matlab then displays a summary of the study:

SUMMARY:

Micrograph studied: 23_SEM.TIF, size of the micrograph: 400 x 600.

Resolution of one pixel: 0.314 µm x 0.314 µm.

Number of pores: 617, for a filtered porosity of 43.63 %.

Overall connectivity for this micrograph: 0.76 %.

Compared to an apparent porosity at the first evaluation: 52.53 %.

A final step to this study is to provide a tool to study every micrographs as precisely as possible. For this purpose, each time the program is run; a file is created with the property to append every further result, saving both the name of the micrograph studied and the filtered porosity.

A statistical study is then computed to calculate the overall mean and standard deviation for a given sample name, i.e. 1_SEM, 23_SEM or 74_SEM without the extensions in order to get a value per sample and conclude about the porosity per case of study.

Application and Results

Cases of study:

Table 4: Image analysis cases of study

case	1	2	3	4	5	6
temperature	25°C			300°C		
amount of starch	0%	10%	20%	0%	10%	20%

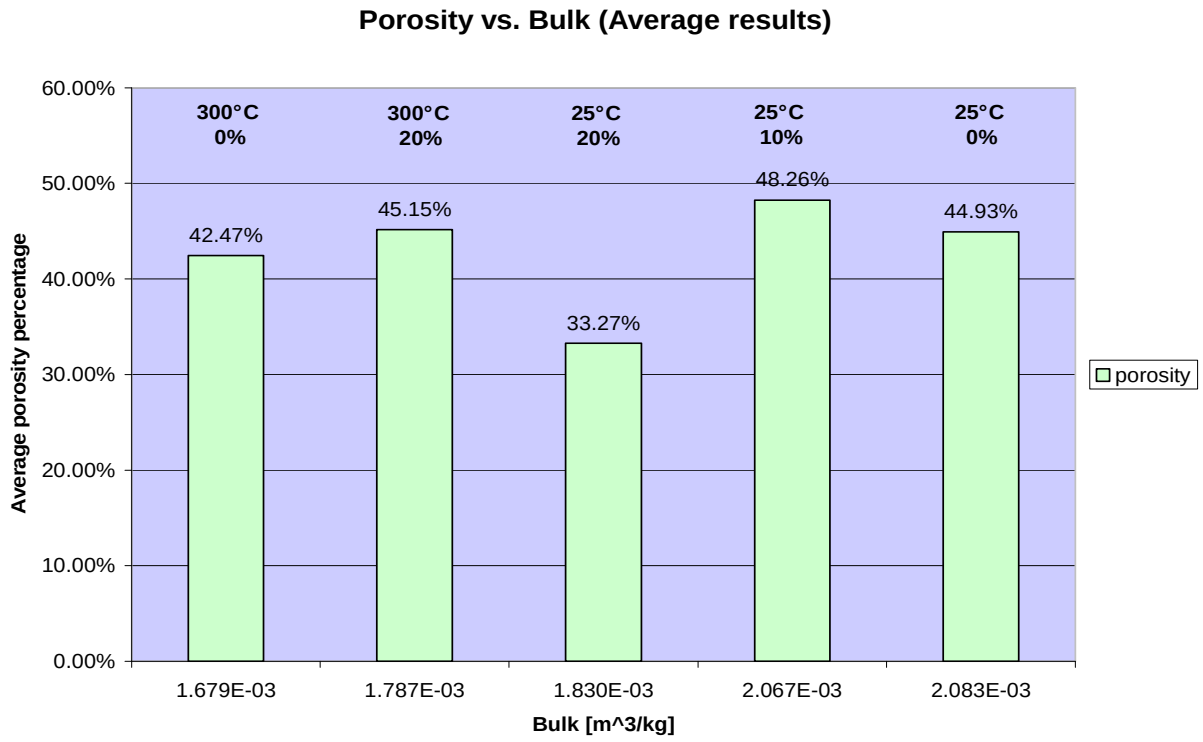


Figure 14: Results of porosity analysis

The results above are to be taken as a trend. The image analysis program is in current improvement, and the version presented here is useful in giving a basis of comparison on various micrographs in terms of average. As we can observe here, there is an interesting trend in terms of porosity between the different cases of temperature.

When dealing with the process of wet pressing, we can observe a sharp decrease in porosity when the highest amount of starch is used. These results are based on a fair

amount of analysis, so the average can be trusted. The goal is to cross check porosity results with the hypothesis of bulking effect in starch imbued impulse dried handsheets. Unfortunately, no micrographs are available for our most interesting case at this time. Our expectation goes to the case of 10% relative content of starch in handsheets impulse dried at 300°C. The availability and conditions of the S.E.M. were an issue at that time. However, we can already observe that the overall porosity is somewhat preserved, which means that impacting the paper web structure with starch doesn't influence so much the porous structure in terms of porosity (except for the case of wet pressing with 20% of relative amount of starch which is definitely not a desirable case). We have thus to investigate further on the impact of such a procedure.

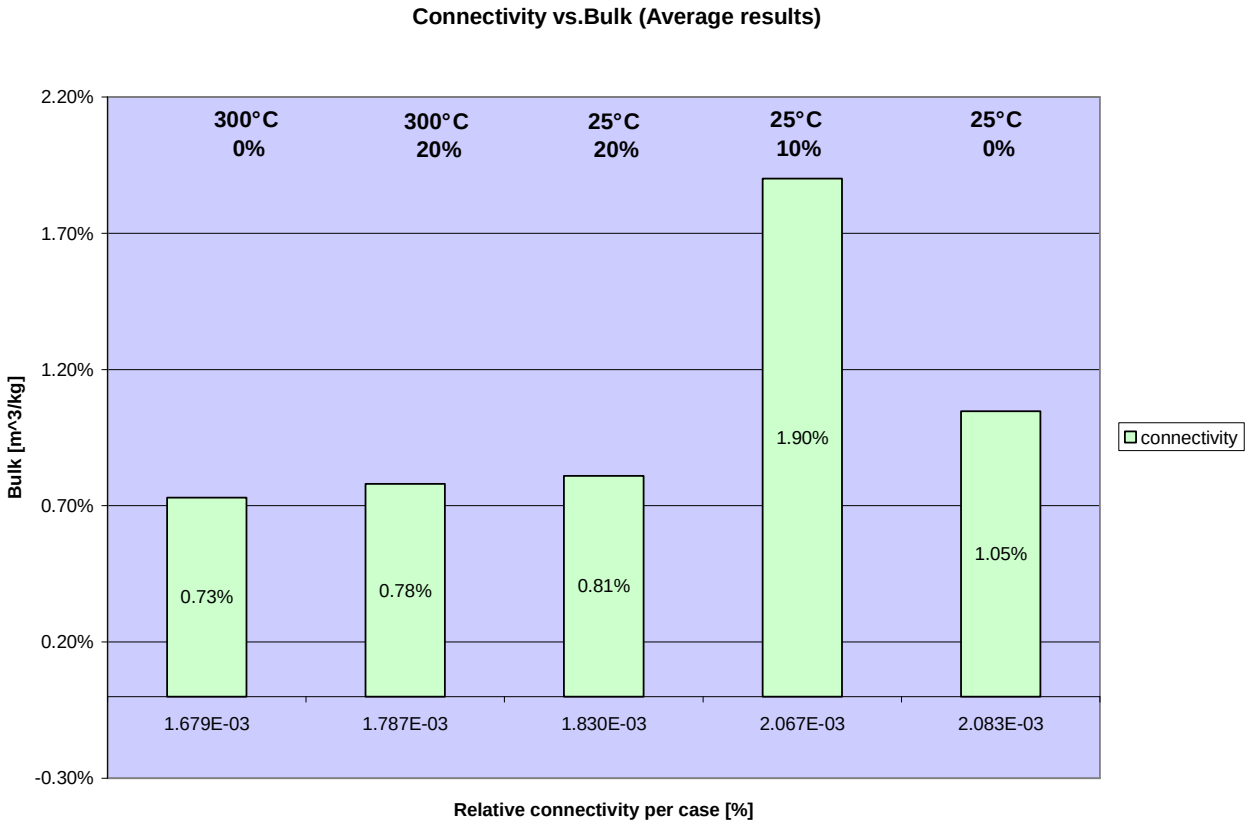


Figure 15: Relative connectivity

The relative connectivity study has also its range of interest. It is defined as the percentage of the porous structure that is connected between each other. A simple (although limiting) criterion is defined as a portion of 6 by 10 pixels of porous structure between two distinct pores. As we can see, there is a neat tendency for closed porous matrix in the case of starch imbued handsheets. However, here again our case of 10% relative content of starch in handsheets impulse dried at 300°C are still expected.
Over the number of simulation runs, we can fairly conclude on the impact of starch use in a paper handsheet. It is clear that the use of starch allows a less open structure in the web. As we have seen before, the overall porosity is preserved, however, as we can see now, the pores are less connected to each other. We can conclude that water during impulse drying especially is to be trapped in these closed pores, hence a possibility of bulking which is not encountered during wet pressing since the flash evaporation process does not occur. However, we still don't know how this bulking effect occurs. All in all, we can only conclude on a visible trend without being able to confirm anything at this point.

The study of pore size repartition is a very good tool to study the structure and bring conclusion on the performance of impulse drying on starch imbued handsheets. For this study, we're going to focus on the extreme case: wet pressing on control case handsheets and handsheets with 20% of relative amount of starch, as well as impulse dried control case and 20% of relative amount of starch. Again, results are presented as an average over several samples per case.

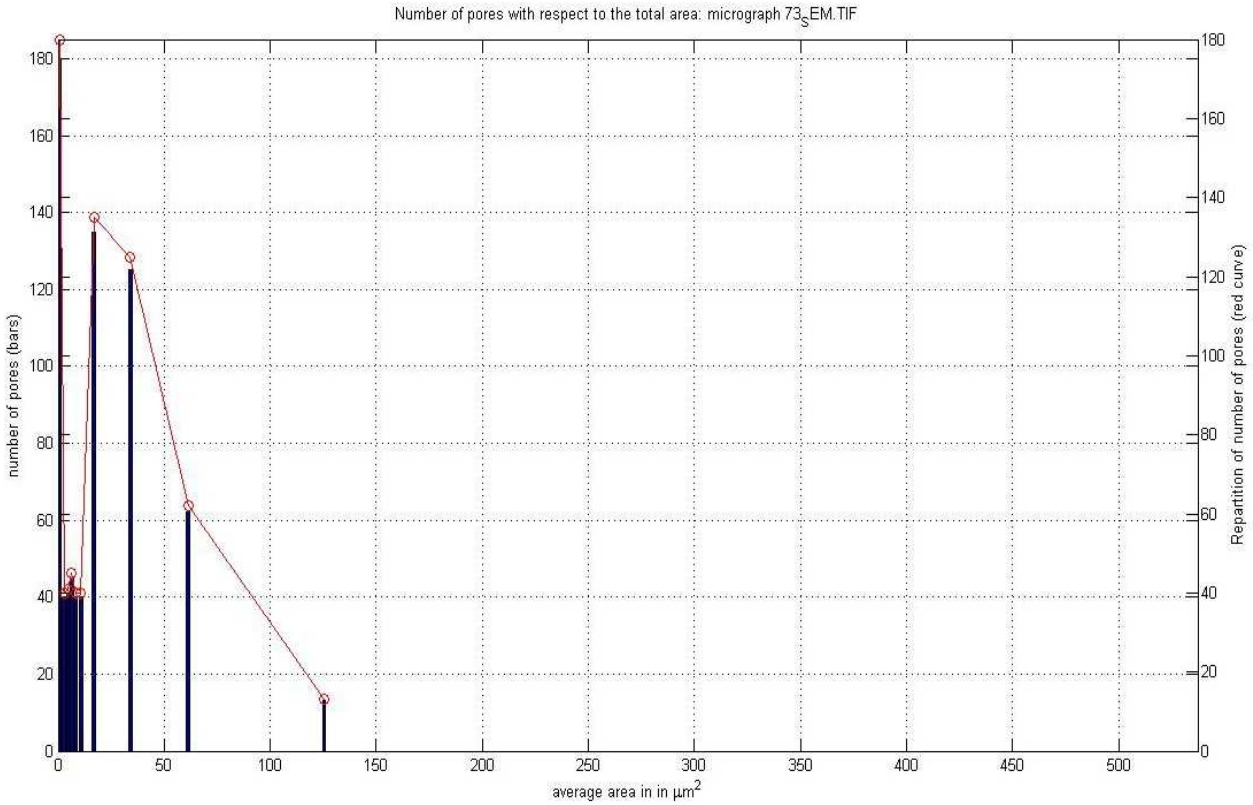


Figure 16: Pore size distribution for a control handsheet pressed at 25°C

On this figure, bars represent the number of pores per category as defined earlier. Again each category is defined with respect to the pore with the maximum size. The last bar represents the number of pores with a size between 50 and 100% of the maximum size, the bar before the number of pores having a size in between 25 to 50% of the maximum size and so on… We can clearly see that in the conditions of wet pressing for a regular handsheet, the structure is essentially microporous. Only a few "big" pores are obtained, having a size less than a 125 µm². No delamination is usually encountered in these conditions of experiments although the web shows the highest bulk from ultrasonic testing, but it shows also the worst case of out-of-plane elastic modulus.

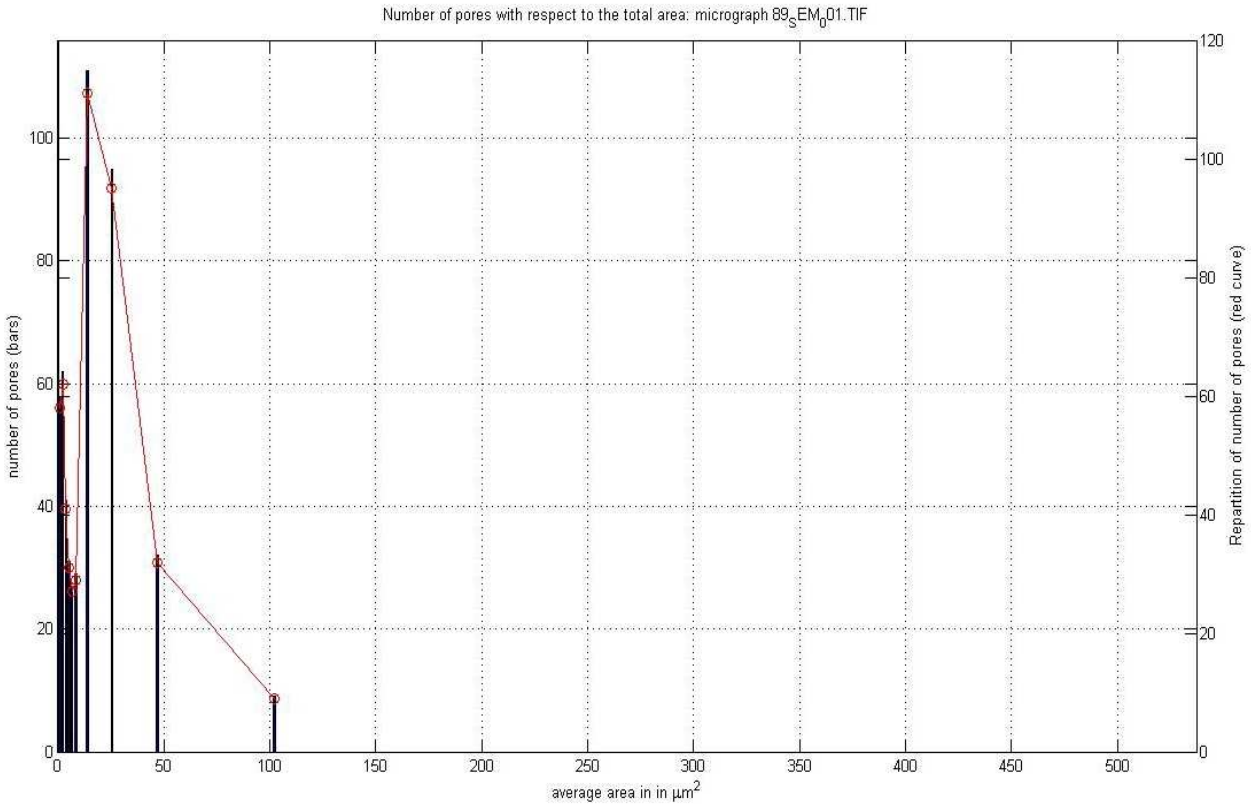


Figure 17: Pore size distribution for a 20% handsheet pressed at 25°C

On this figure, we can observe the effect of starch in a wet pressed handsheet. As these results are taken from various averaging, we can see that starch is impacting the structure on the pore size distribution. Here, we have a slightly chance to find pores above 100 µm². We can recall from previous studies that wet pressed starch imbued handsheets show less porosity than any other wet pressed handsheets. This clearly shows the fact that a wet pressed starch imbued handsheets is densifying a lot under the action of starch as a strengthening agent, hence a well bonded dense web (But one of our worst case in terms of bulk anyway).

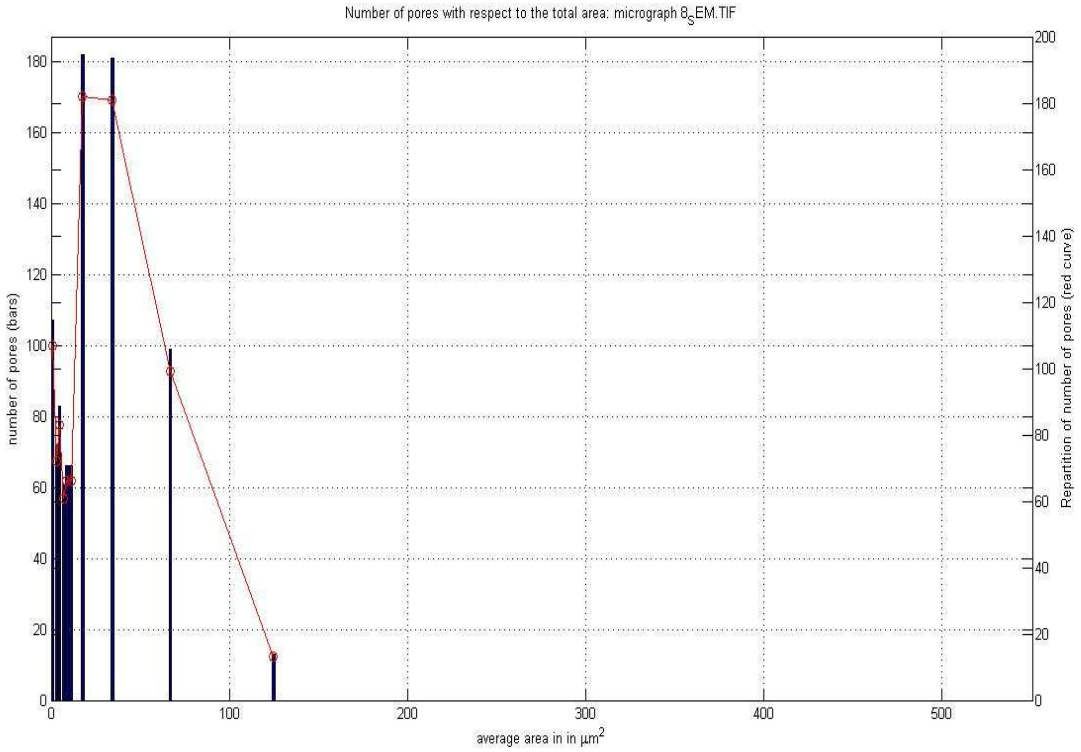


Figure 18: Pore size distribution for a control handsheet pressed at 300°C

Control handsheets impulse dried handsheets show a somewhat improvement in the pore size distribution. The web exhibit more properties of a “bigger” pore distribution in the sense that most of the pores are found in between 20 to 100% of the maximum pore size encountered during the analysis. As opposed to the wet pressed case where the majority of the pores are to be found in the “micro” range under 10µm², this case shows a good uniformity in terms of porosity. However, bulking is definitely not reached. This case is the worst to be encountered on both parameters: bulk and strength.

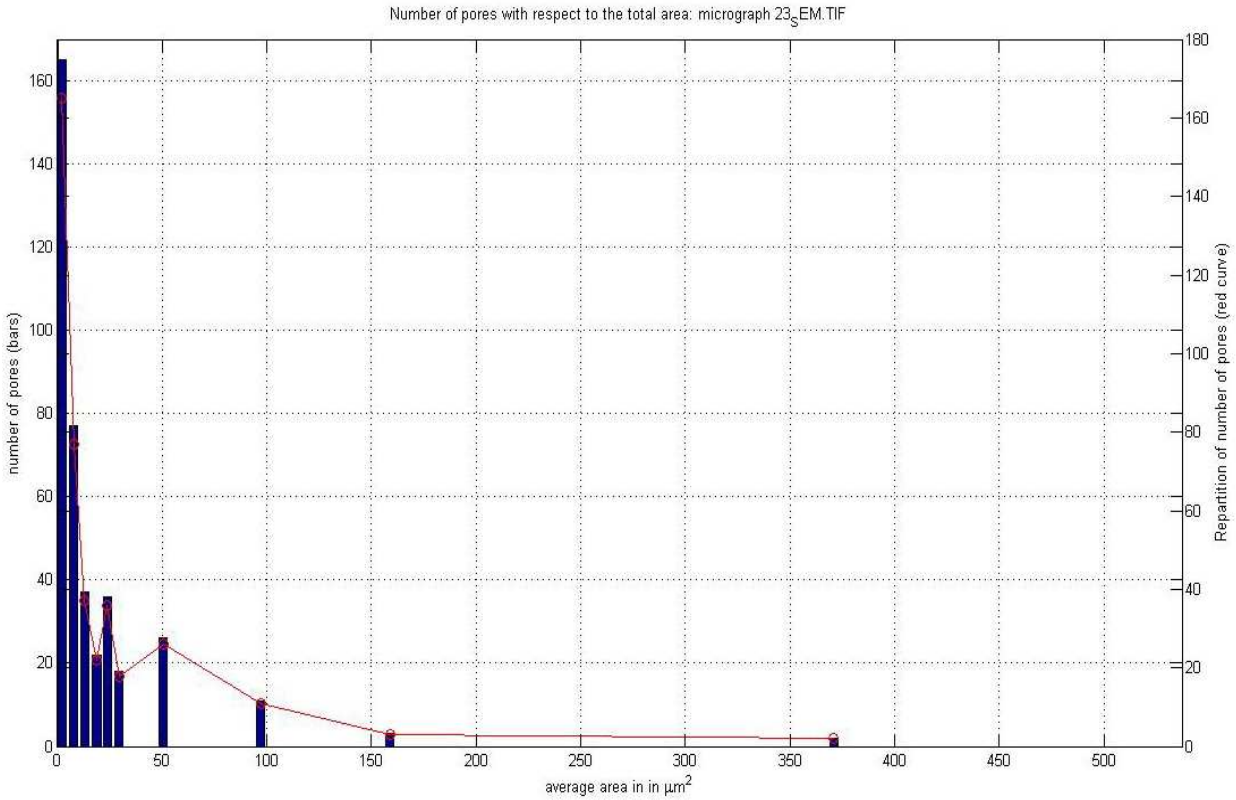


Figure 19: Pore size distribution for a 20% handsheet pressed at 300°C

In the case of starch imbued handsheets impulse dried at 300°C, there is clearly a big difference compared to the three other case. We know that the global porosity is preserved all in all; we know also that there is significantly less connectivity. And as we can observe on the pore size repartition, we find some pores almost 4 times bigger than the other case. We clearly have here macropores at a range in between 350 and 400 µm². The fact that there are several pores of this size points out one important fact: we are able to control delamination using starch in combination with high impulse drying temperature. Hence, as we can observe on several micrographs in this particular case, we don't create a foamy structure out of the amount of starch, but we clearly reinforce the web up to a point that the overall structure is closed enough to trap water. This has the effect to start delamination during the flashing process. However, since the fiber mat is reinforced by starch (fibers are clearly coated by starch when dealing with bleached

fibers), delamination is stopped and as a result, macropores are created as a consequence of the flashing evaporation and high internal pressure contained.

We have clearly reached our goal here, and surprisingly our conclusion lead to a completely different explanation than the one formulated by our working hypothesis. We don’t create a foamy structure, neither obtain a bulky structure thanks to the capability of starch to expand, but we contain delamination before the web rip apart, hence creating macropores in the structure that account for the bulking effect during impulse drying at high temperature on starch imbued handsheets.

Following is another summary of porosity and connectivity along with the standard deviation after several simulation run.

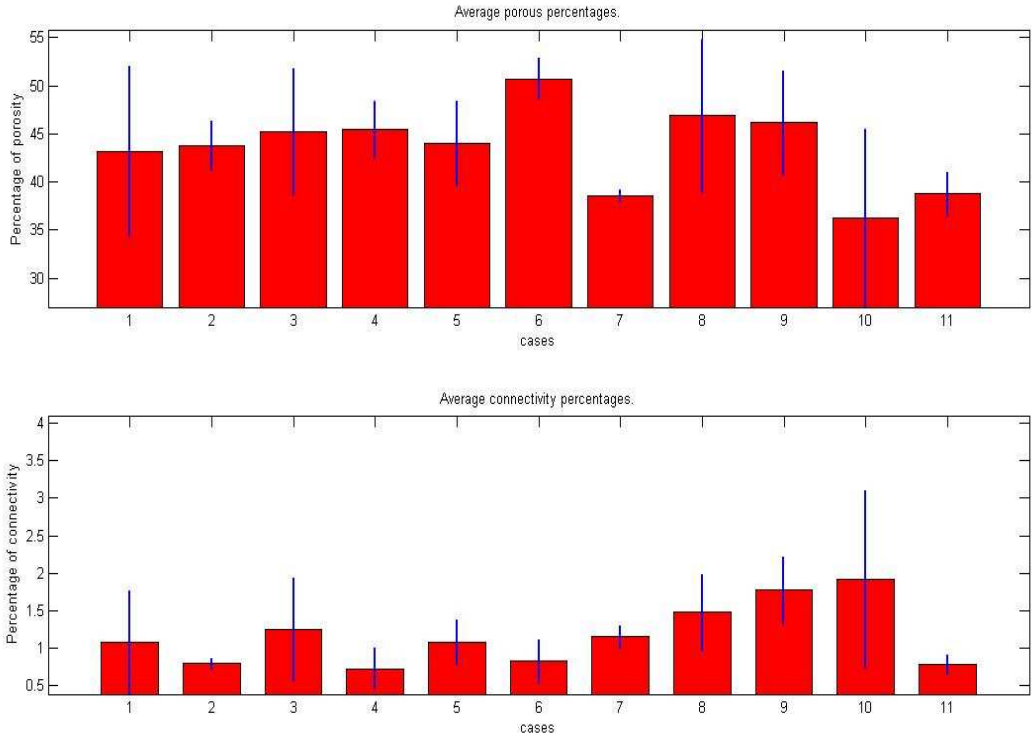


Figure 20: Summary of porosity and connectivity percentages for all cases

Cases	1	2	3	4
micrograph	1_SEM	8_SEM	19_SEM	23_SEM
starch content	0%	0%	20%	20%
temperature	300℃			

5	6	7	8	9	10	11
73_SEM	74_SEM	75_SEM	85_SEM	88_SEM	89_SEM	90_SEM
0%	0%	0%	10%	10%	20%	20%
25℃						

CHAPTER 6

CONCLUSIONS, FURTHER INTERESTS AND STUDIES

On Impulse Drying and the Physics of Process

Impulse drying is known since a few years for its performances as a complementary tool to the dry end section. It is also appealing for its potential in terms of energy saving. Yet there are still some issues that prevent its usage in the industry. In an effort to optimize a step further its promising use, the physics behind this process have been early addressed in order to target the range of actions to take.

As a consequence, the process of impulse drying is known now to be sequenced in four main time intervals. The first one is a classical wet pressing process which impacts the paper web by reducing the pore volume, hence triggering dewatering. The second time interval is known as the start of boiling pool heat transfer during which a two-phase flow starts in the porous structure, thanks to the combination of heat transfer from the hot platen and increasing internal pressure. During the third time interval, controlled flash evaporation starts and is the key parameter in impulse drying, in terms of performance. During this time interval, bonded water starts to vaporize, thus enhancing dewatering performance. The last time interval happens at the nip opening, where external pressure drops to zero, which let the process of flash evaporation continuing uncontrolled. While these time intervals are well defined, and characterize fairly well the processes of heat and mass transports, only heat transfer process has been extensively analyzed in the early 80’s. This first step has led to numerous models which in turn have helped understanding a little bit more the impacts on the paper web structure.

From this stem point, the question of paper web properties has been an issue in models accuracy and the necessity to define rather limitating assumptions and/or simplifications. However, important conclusions have been expressed in terms of the impact of two-phase

flow in the porous structure, leading to hypothesis regarding rewet reduction, vapor front impact on delamination, internal pressure model etc. This has been the opportunity for a new series of experiments concerning impulse drying improvement in terms of technology.

A second big trend concerning impulse drying concerns these efforts to actually improve the control of impulse drying and come up with a large scale prototype. This has given the opportunity to study several factors on drying performance and paper quality at the end of the drying process. Hence, several improvements have been possible especially concerning the control of heat transfer by using low-thermal mass coating material for the heated roll, leading to conclusion on critical impulse temperature on delamination, and a series of criterion on the role of external pressure on the process performance as well. Meanwhile, several paper properties such as thermal softening, densification, and surface properties have been investigated to characterize the advantages of impulse drying on paper quality. This has leaded more and more to the fact that impact of furnish quality, basis weight, ingoing solids, and the porous structure had to be addressed more precisely.

On Experiments and Use of Starch

As the study on impulse drying technology in itself starts to be quite exhaustive, it is only logical to turn on the product itself, namely the paper web, in order to investigate the parameters of performance with regards to the structure. As early experiments have shown, one major issue concerning impulse drying is the problem of delamination, which is believed to be the consequence of unrestrained flash evaporation, leading to internal pressure too important for the fibers bonding. Hence a first series of experiments have been performed in late 2005 to start investigating the relative strength of wet paper web as well as analyzing the parameters on paper web strength. For this purpose, a series of experiments have been designed to interact with the inner structure of the web. Since the

technology is fairly understood, and parameters quite controlled, it is the opportunity to use its specificity while acting directly on the paper structure. In this order of idea, Dr. Orloff came up with the idea of using starch to a relative high extent in the paper in order to affect the out-of-plane strength, while taking advantage of an available product with promising properties. Hence, the fact that starch is used in the paper industry as a retaining and strengthening agent, combined with its capability of expansion under high temperature makes it a perfect candidate to further take advantage of impulse drying technology.

This thesis has been the occasion to pursue these efforts, and analyze thoroughly the parameters of out-of-plane strength along with bulking of the web in various conditions of temperatures and relative starch content. After an analysis of various type of starches in order to determine the optimal choice, the impact of temperature on paper strength has been studied. It has leaded to promising results both in terms of elastic modulus and bulking effect. At this state of study, it seems that both desired effects can be achieved by the combination of impulse drying and use of starch. To the amount of starch used, it is then fairly promising in terms of impulse drying performance. This means that not only delamination is about to be controlled, but also the use of raw material could be reduced (not to say that impulse drying allows the use of low quality furnish with very good results in terms of paper quality).

Here again, these results have leaded to another perspective of analysis. In an effort to understand deeper the process of delamination as well as the impact of starch on the paper structure, hypothesis have been formulated concerning the process of starch integration. Hence, it is believed that the use of starch leads to a reduction of pore connectivity; in other words, a closed fiber matrix which prevent venting and possibly restrained the later part of flash evaporation. The latter is also believed to trigger the bulking effect observed during impulse drying with starch imbued handsheets. A model

of pore expansion has been formulated in an effort to model the phenomenon occurring in such experiments. As a consequence, the next step of this thesis was to come up with a tool to observe the porous structure, in order to determine the input parameters for this model and get more material to support or contradict the formulated hypothesis concerning pore connectivity. In parallel, a few experiments have been designed in order to investigate the quality of starch integration in the handsheets, which turned to be a complicated task.

On Porosity and Delamination

As the question of porosity is raised, it often comes with the problem of transport phenomenon and especially with paper drying, mass transport in place of dewatering. A big trend in the late 90's has been toward the model of multi-phase flow, which is of great interest concerning the drying section and the performance of dewatering. Here again several approaches have leaded to simple models with several assumptions. An opportunity to understand more closely the transport phenomenon in a porous structure such as paper web has arisen, with the concern of paper web behavior during the drying process, especially under such high intensity process as impulse drying. The formulation of a two-phase flow model with the combination of laws such as Darcy's law or Fick's law have permitted a better understanding of paper properties such as relative permeability, the importance of macropores, the issue of paper mat compressibility and various parameters such as heat of sorption, viscosities etc. This has raised the question of specific parameters in paper and fiber such as fiber densities, capillarity, wicking, thermal softening, etc.

Meanwhile, a growing interest on the porous structure has guided several experiments. The main interest is, for the case of drying, how to make the best use of a porous structure as far as dewatering is concerned; while of course avoiding the lost of paper

quality. Hence, transport models are more and more coupled with the characteristics of the porous medium. It is clear now that further steps are to be taken in complexity if one is to approach and optimize dewatering in the paper industry on a general point of view, but especially more when concerned by the potential of impulse drying. In that spirit, the fully detailed heat transports process have been integrated to mass transport models in porous medium, giving insights to a significant number of parameters affecting the process performance. The first impact has been on a better understanding of fiber mat behavior and the emergence of stochastic studies to try to characterize the flow in a porous structure. Numerous techniques, based on intrusion of a phase (either liquid or gas) in the structure have offered a mean to investigate the porosity of paper structure and the phenomenon of capillarity, and to link afterward more precisely internal pressure and sheet permeability on the different time intervals encountered during impulse drying on a transport basis. Some parameters are still presenting some limitations to fully optimize impulse drying, and the use of rheological models is an important step for the future drying. Research have clearly shown that the modification of the porous structure during either pressure events or drying events (and more in case of simultaneous application as it's the case in impulse drying) is impairing in a way or another the potential of dewatering. Concern about the capability of the web to vent out vapor, while resupplying liquid water to heater platen, is of great importance in this case. Comparatively the web capability to resist disruption is still to be addressed with the right operation on the paper structure.

On Image Analysis and Connectivity

The logical step to take when dealing with paper drying and the impact of its porous structure to the overall process is of course porosity examination, along with various other parameters. As described earlier, a significant amount of intrusive methods have been put together to achieve this goal. However, one quickly grasps the limitations of

such methods when dealing with pore-size distribution, which is a major parameter of control on the two-phase flow during impulse drying. Not only the distribution of micro versus macropores is essential on the building of internal pressure and venting of vapor etc. but it is also a significant parameter for the final product quality (either with concerns of printability, opacity, smoothness…). As computing powers are getting more and more reliable, and new computational methods are arising, it is nonetheless important to follow the advances in that area with the new techniques of materials visualization. As described in this thesis, destructive to non destructive methods are available for that purpose, with a growing capacity of magnification, allowing now observation down to the micrometer and even less. The trend is clearly going to a full 3D visualization, but while this goal is not attained, it is essential to keep optimizing 2D approaches. Among the numerous techniques of microscopy, scanning electron microscopy has been chose for our study for its capability of resolution and the rather limited amount of artefacts produced during the imaging process.

Getting micrographs of desired samples is going of course with its analysis. Hence a big part of this thesis dedicated to image analysis, and the specific approach to paper and porosity analysis. It is important to know which parameters are of interests before selecting a specific software or computational method. Again, our goal was to investigate the potential of starch used in a significant amount to avoid delamination. The fact that along the way, qualities such as bulking or pore connectivity became a concern as well really defines the steps to take in terms of computation. For such a goal, it is necessary to come up with software adapted to the analysis of paper web, which can be extremely tortuous with some issues in terms of resolution and contrast differentiation. Therefore an approach first on thresholding, leading gradually to the study of edge detection methods was logical. As we are working on a process dedicated to energy savings in the paper industry, it is also essential to keep in mind that the same goal is to be applied on any of

the steps of study. That is in terms of computation an economy of calculation as well as an economy of storage capacity. The image analysis program has been created in that particular purpose.

Interestingly enough, the results of image analysis are tending to prove that the hypothesis of closed paper matrix is valid in the case of starch imbued handsheets, although some more experiments have to be performed as well as more image analysis run to fully clarify this trend. This proves at least one thing, numerical simulations are here to complement and to orient research on sets of experiments to grasp a concept standing behind a physical phenomenon.

Integrating to Global Research

A big concept to a real necessity: more than the various conclusions brought by the subject is the potential behind. Along this theme of research, I come to realize the field of paper science is rich in promises, and is open to numerous improvements. However, it is not an easy task to reach that goal. As a matter of fact, studies have been so intensively sought by the industry that somewhere, simplifications become a liability. Actually, this area is facing now its own clarity about various phenomena that these become a complexity in itself.

As far as impulse drying is concerned, the technology is fully understood, the mechanics behind drying is almost completely understood; but surprisingly enough, both sides of these improvements are not yet put together. That’s really where the area of paper science has to go now. It is impossible, or rather meaningless, to keep studying the process of heat transfer for its own sake while we know its correlation to mass transport, knowing what we know today. In that spirit, it becomes meaningless, or rather inefficient, to keep studying couple equations of transport while trying to grasp separately the impact of

porosity on the flow (meaning various concepts such as capillarity, wicking, tortuosity…). Further more, it is inefficient, or rather sad, to see that the particular impact of fiber properties in this porous medium is not yet seriously integrated to the previous areas for the sake of drying improvement in paper related areas. And so on and so forth. Going quickly back to the numerous models exposed in this thesis which are related to the physics of drying, we should now understand the importance of integrating heat and mass transport to porous materials flows and rheology. If another step is to be taken in this understanding, paper science cannot afford any more simple models while they're already limited by numerous assumptions that should clearly be addressed.

Again, in a same trend, the big picture is to be taken in all the areas. One cannot anymore concentrate on experimental approach while ignoring a numerical simulation of that very approach, and forgetting the basis of comparison between both of them. One doesn't go without the other, and experiments have to be formulated based on the complex physics of drying (talking about impulse drying), which will bring materials to formulate a simulation, which has the purpose of comparison with various external methods for the sake of feedback, and more experiments until a commercial concept or a full understanding support is reached.

Back to our subject, it was clear from the beginning that impulse drying technology has everything to gain from serious improvements. In this sight line, it was clear that addressing delamination is a priority. How then can we come up with the next step to take? Well, once studies are reviewed, it is clear that lots of efforts have been put on the technology itself, while almost forgetting the material of interest: paper. Hence, the idea is to concentrate on paper web, keeping in mind strength is the factor to study. Once an idea such as the extensive use of starch is formulated, one shouldn't make the mistake to keep focusing on the area, but 'au contraire' one should start to broaden the idea and the

approach. With this perspective, it is now clear that such a subject couldn't have been approached without combining experiments with a model formulation and the study of imaging techniques concerning the paper web, in parallel with the necessary image analysis. This goes as far as broadening the research to various areas such as heat and mass transfer, porous materials study, use of polymers and their properties, computing and imaging techniques, basic chemistry to treat samples in various ways, measuring techniques such as ultrasonic testing and microscale approaches, to complete with numerical simulation and a minimum of statistical mechanics. There stands the reality of research in paper science and the concept of global research.

A necessity of cooperation and broad understanding face nowadays most of engineering projects. It is particularly true with paper science which mixes such areas of study as mechanical engineering, chemical engineering, biology, material science, applied mathematics, computer science and any micro/nanoscale areas.

As far as the future goes for impulse drying, there is truly a potential with regards to the use of starch with the paper web to the extent that the fibers are coated with this polymer. The measurements have shown the existence of this potential, the image analysis have put a step closer to a proof, while there are still some efforts in terms of optimization that are already in progress with the use of edge detection. The existence of a math model for pore expansion is a key element to further the study of starch potential, as well as more generally the fiber mat behavior under pressure constraints, as well as temperature constraints once starch characteristics will be included. It is clear that efforts are to be put in understanding the relative compressibility of the fiber mat, with or without any additives such as starches.

The study of micrographs presented in this thesis goes already beyond the simple study of porosity and relative connectivity of pores in the paper web after impulse drying process

(or any other drying process). It is known that wet samples can be treated for scanning electron microscopy, and this was the next step to be taken for this project, as a tool to measure initial parameters prior to drying process. These parameters were intended as input parameters for the numerical study of pore expansion. However, the lack of funding and knowledge in the preparation of wet samples has slowed down this effort which was crucial in order to test the math model.

However, some further steps have already been taken regarding again image analysis. Micrographs coupled with edge detection offer another wonderful opportunity in terms of numerical analysis. One has here a perfect material for finite element analysis. Detecting edges between pores and fibers offer the advantages to have at hand boundaries for a finite difference/element/volume study. Having at disposition various mass and heat transport models that can be coupled with relative fiber mat compressibility offers the possibility of discretization while the micrographs can easily be meshed for a numerical study. It will be easy to used edges as starting point to mesh afterwards the porous structure, with an adaptative mesh size depending on the size of the pores, the connectivity and capillarity of the material. Having those at hand, it is a nice idea to make use then of the model of pore expansion in order to predict the fiber mat behavior starting with micrographs of wet samples. All these predictions can then be compared with impulse dried samples, and that's where a rheological model has all its importance. There is here a real opportunity to take a significant step toward impulse drying improvement while offering to the paper industry a future statistical tool for paper density/bulk prediction.

In parallel, more studies of starch coated fibers have to be performed to grasp the main parameters of such a system, here again in terms of compressibility, expansivity and other material properties. This combination has already shown some promises, but a further step is again possible if one was to consider baking soda as a complement in terms

of expansion. There is no reason to limit oneself with only one “agent” of improvement while others are known to behave in the same trend. A combination of starch and baking soda should prove interesting in many aspects, and a step forward in terms of experimentations. Speaking of which, numerous experiments have still to be performed concerning the behavior of starch imbued handsheets with respect to various conditions of pressure. It is of great importance to understand the role of pressure gradient at the nip opening, since the brief study of pressure variation in wet pressing has shown some curious results. It is to be expected that a real impact will be found with such a study in terms of impulse drying. One cannot ignore the unrestrained flash evaporation process in the fourth time interval of impulse drying; and things are to be investigated on this phenomenon, especially if it could be controlled by internal factors (i.e. in the paper web with additives). The various discussed models have shown some interesting features as well in terms of impulse drying, and properties such as rewet reduction or two-phase front position with respect to the caliper have been suggested. It would of good interest to formalize a series of experiments toward these observations since they have a good probability to be linked to our problem of delamination and our solution of web reinforcement.

Finally, paper science shouldn’t be “afraid” anymore of that infamous area of statistics. Powerful tools in terms of stochastic studies have been formalized by mathematicians, and they’ve proven to be very useful in various areas already. Topics such as Brownian motion should be of great use in the field of drying performance, when dealing with transport. It would be a good idea to start digging extensively in this area of study.

As you can see, a lot is still to do in the particular topic of impulse drying, but interestingly enough, not only for this area. At least in terms of paper science, common

efforts should be put together to “upscale” the understanding of various processes, as a whole with its various background requirements.

I’ve had the occasion to deal with numerous areas as a mechanical engineer, and I’ve realized that the area of paper science lacks of multi-skilled approaches. One cannot be uniquely a mechanical engineer, or a chemist, or a biologist … But all of the above and more! That’s where solutions are and the future of this science in itself, namely paper science. That’s the major point I’ve learnt from Dr. Orloff, would I be to leave with just one take away … which is not the case.

APPENDIX A

PREPARATION OF EPOXY SAMPLES

Procedure

Preparation of Epoxy Resin:

The hardest formulation of Spurr resin is recommended for embedding. Use the available kit containing for bottles: “Low Viscosity Embedding Kit” Cat. #14300

ERL 4206 (VCD): 9.2 ml
DER 736 : 5.5 ml
NSA : 25.5 ml
DMAE : 0.3 ml

Caution:

VCD is an animal carcinogen, toxic and corrosive.
NSA causes skin and eye irritation.
DMAE is corrosive.
DER causes skin and eye irritation.
The chemicals used for the epoxy resin preparation, especially DMP-30, are toxic and they cause skin irritation.
Plastic gloves are required to handle these chemicals. In case these chemicals come in contact with your skin, they should be washed away completely with soap.
Spurr resin has to be mixed in fume hood.

Use four different plastic pipettes to dose the different chemicals.
Pour ingredients into a plastic beaker (100 ml) and mix them vigorously for 5 min with a wood stick.

Embedding

Fill the mold with the resin and de-gas sample, first in fume hood to let epoxy penetrate the paper samples during 15 min, then in a vacuum chamber until trapped air bubbles are completely removed from the samples (~3 to 5 min), vacuum up to 25 mm Hg.
Cure the resin in an oven at 70°C for 2 days until the resin becomes hard enough. Check the sample after 2 hours to see if there are any leaks, if so pour some more resin.

Polishing

Take out the embedded sample from the mold (using hammer for example).
Drill a hole at the bottom of the sample and thread in order to put a screw in it, which will help to hold the sample during etching.

Coarse Grinding:

Set 120 grit (3M) abrasive paper into the grinding machine (Buehler Ecomet 5) and grind the block surface at 240 RPM. Remove epoxy resin until the whole paper samples cross section appear on the surface.

Edge of the paper samples are damaged when cut, so remove the damaged edge of the samples by coarse grinding.

Clean the block surface with a lint-free cloth to remove coarse grains. Clean the grinding machine to prevent contamination by coarse grains.

Set 320 grit (Struers) abrasive paper and grind the block at 240RPM.

Clean again the block surface.

Set 800 grit (Struers) abrasive paper and grind the block at 240RPM.

Clean the block surface.

Fine Grinding:

Set 1200 grit abrasive paper and grind by hand gently. Clean the block surface.

Set 2400 grit abrasive paper and grind by hand gently. Clean the block surface.

Set 4000 grit abrasive paper and grind by hand gently. Clean the block surface.

Each grinding stage should take about 15s to 2 min.

Preparation of solvent

Take a 125 ml beaker and add 4 grams of Potassium Hydroxide (KOH) pellets to 20 ml of 100% methyl alcohol (CH_3OH) and 10 ml of propylene oxide (C_3H_6O).

Some heat is generated during the dissolution. Cooling the mixture is required to prevent boiling.

Stir during 20 min in order to dissolve potassium completely.

This solvent should be prepared just before use.

Etching

Insert a rod to hold the block.

Immerse the block into freshly prepared solvent. The block is immersed just deep enough to keep the surface wetted.

The rod is twirled to provide enough shear and continuous supply of solvent at the surface.

10 µm depth of removal is adequate to reveal specimen structure (1 to 2 min treatment).

Wash the block surface by 100% methyl alcohol three times and let the block dry (use vacuum).

Coating

The samples have then to be gold coated in order to make them conductive for electrons, without which, samples would simply burn and provide incorrect information for the analysis.

APPENDIX B

MATLAB: DETAILS AND CODE

Summary of **imAnalysis** functionality

- ✓ This function takes 4 arguments: micrograph's filename; size of a pixel, height of the micrograph to study, width of the micrograph to study.
- ✓ It first reads the image and performs contrast equalization. Results are provided temporarily in Matlab figures.
- ✓ A threshold is then determined based on the best equalization with respect to our study of pores versus fibers.
- ✓ A criterion of connectivity also has been determined based on a study of several micrographs.
- ✓ A quick set of information is displayed on screen in order to give information on a rough estimation of porosity.
- ✓ For record, an illustration of modified micrograph to put evidence on fibers against the possible pores is saved in a file called Pores_"FileName".
- ✓ A Word document is created to keep a record of every study about porosity, thus saving also the most probable range of intensities of the studied micrograph as well as the threshold.
- ✓ Finally, the workspace is saved to offer the possibility to get access to any variables further on. Typically the workspace is saved under the name "Workspace*Name_of_Micrograph*"

Summary of **bkPix** functionality

- ✓ It takes in input the previous Workspace.
- ✓ Run the count of black pixels encountered while scanning lines of the contrast modified micrograph.

- ✓ Save in an array all information accessible about these pixels.
- ✓ Display some information, among which the running time since it might take a few minutes on high precision micrographs to run the process.

Save the workspace in a new variable called "Black*Name_of_Micrograph*"

Summary of **filtering** functionality

- ✓ Work on the colored image containing red pixels which stand for possible connectivity under the tolerance criterion.
- ✓ Proceed line by line to reconstruct both pores and fibers as accurately as possible.
- ✓ Save the workspace to be used in the next step of the analysis.
- ✓ Illustrate the result of filtering in a new figure.

Printed results using Matlab

Below is an example of these results which are saved in a text file:

```
*  *   *   Results for the micrograph: 1_SEM. *   *   *

Average porosity: 52.66%.
      Standard Deviation: 9.58.
Average connectivity for this sample: 0.54%.
      Standard Deviation: 0.17.

*  *   *   Results for the micrograph: 8_SEM. *   *   *

Average porosity: 42.01%.
      Standard Deviation: 1.89.
Average connectivity for this sample: 0.76%.
      Standard Deviation: 0.02.

*  *   *   Results for the micrograph: 19_SEM. *   *   *

Average porosity: 39.98%.
      Standard Deviation: 0.00.
Average connectivity for this sample: 0.52%.
      Standard Deviation: 0.00.

*  *   *   Results for the micrograph: 23_SEM. *   *   *
```

```
Average porosity: 45.15%.
      Standard Deviation: 1.82.
Average connectivity for this sample: 0.78%.
      Standard Deviation: 0.35.

*  *   *   Results for the micrograph: 73_SEM. *   *   *

Average porosity: 45.42%.
      Standard Deviation: 4.36.
Average connectivity for this sample: 1.14%.
      Standard Deviation: 0.33.

*  *   *   Results for the micrograph: 74_SEM. *   *   *

Average porosity: 50.41%.
      Standard Deviation: 2.50.
Average connectivity for this sample: 0.77%.
      Standard Deviation: 0.33.

*  *   *   Results for the micrograph: 75_SEM. *   *   *

Average porosity: 38.59%.
      Standard Deviation: 0.61.
Average connectivity for this sample: 1.15%.
      Standard Deviation: 0.15.

*  *   *   Results for the micrograph: 85_SEM. *   *   *

Average porosity: 48.34%.
      Standard Deviation: 8.38.
Average connectivity for this sample: 1.57%.
      Standard Deviation: 0.52.

*  *   *   Results for the micrograph: 88_SEM. *   *   *

Average porosity: 47.13%.
      Standard Deviation: 1.15.
Average connectivity for this sample: 1.67%.
      Standard Deviation: 0.49.

*  *   *   Results for the micrograph: 89_SEM. *   *   *

Average porosity: 37.78%.
      Standard Deviation: 9.44.
Average connectivity for this sample: 2.15%.
      Standard Deviation: 1.16.

*  *   *   Results for the micrograph: 90_SEM. *   *   *

Average porosity: 38.56%.
      Standard Deviation: 2.89.
Average connectivity for this sample: 0.70%.
      Standard Deviation: 0.08.
```

For more information on the micrographs, please refer to the details provided in the table.

Table 5: Details on micrographs used for image analysis

impulse drying temperature	Amount of starch	Sample label	impulse drying temperature	Amount of starch	Sample label
25℃	0%	73_SEM	300℃	0%	1_SEM
		74_SEM			2_SEM
		75_SEM			3_SEM
		76_SEM			4_SEM
		77_SEM			5_SEM
		78_SEM			6_SEM
		79_SEM			7_SEM
		80_SEM			8_SEM
	10%	81_SEM		10%	9_SEM
		82_SEM			10_SEM
		83_SEM			11_SEM
		84_SEM			12_SEM
		85_SEM			13_SEM
		86_SEM			14_SEM
		87_SEM			15_SEM
		88_SEM			16_SEM
	20%	89_SEM		20%	17_SEM
		90_SEM			18_SEM
		91_SEM			19_SEM
		92_SEM			20_SEM
		93_SEM			21_SEM
		94_SEM			22_SEM
		95_SEM			23_SEM
		96_SEM			24_SEM

Matlab Code

```
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%
%%%%%%%%                      Main Program
%%%%%%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%

clear, close all;
warning off all;

%This program runs the subroutines to analyze micrographs in several
steps.
%1- Run imAnalysis to offer the opportunity to choose the micrographs
%2- Save the Workspace in order to get access to the variables for the
next
%   step which is: rough estimation of apparent pores.
%3- Run the calculation of each pores, giving their total area and the
%   number of pores in the micrograph in order to get a statistical
%   repartition of the porosity per cases of study.

%%%%%%%%%%%%%%%%%%%%%
%Program Run time
t1 = cputime;
%-----------------------------------------------------------------------
----
%%
%Read the file containing informations of each micrographs:
%Create Arrays containing name of the micrographs, their resolution,
the
%number of pixels along X and Y:

FileName = char([]); Mag = char([]); Xnum = char([]); Ynum = char([]);
fid = fopen('detailMicrographs.txt','r');
count = 1;
while ~feof(fid)
   j=1;p=1;q=1;t=1;
   line = fgetl(fid);
   if isempty(line)||(line(1)=='F'), continue, end
   k = strfind(line, 'TIF');
   for i=1:k-2
        FileName(count,j)=line(i);
        j = j+1;
   end
   clear('i');
   for i=k+4:k+8
       Mag(count,p)=line(i);
       p = p+1;
   end
   clear('i');
   for i=k+10:k+12
       Xnum(count,q)=line(i);
       q = q+1;
```

```
    end
    clear('i');
    for i=k+14:length(line)
        Ynum(count,t)=line(i);
        t = t+1;
    end
    clear('k','i');
    count = count + 1;
end
fclose(fid);
clear('fid','p','q','t','line','count');
%-------------------------------------------------------------------------
----

%Create an array containing:
% 1- micrograph name -> FileName(i,:)
% 2- pixel resolution
% 3- heigth -> Xpix
% 4- width -> Ypix
% NOTE: using here str2double to be faster, can use as an alternate
str2num
% however less fast.
resolution = []; height = []; width = [];
for i=1:length(Mag(:,1))
    resolution(i,1)=str2double(Mag(i,:));
end
for i=1:length(Xnum(:,1))
    height(i,1)=str2double(Xnum(i,:));
end
for i=1:length(Ynum(:,1))
    width(i,1)=str2double(Ynum(i,:));
end
clear('Mag','Xnum','Ynum');

%-------------------------------------------------------------------------
----
%Choice of micrograph:
disp(sprintf('List of Micrographs:\tResolution: '));
LFN = length(FileName(:,1));
for i=1:LFN
    micrograph = FileName(i,:);
    res = resolution(i,:);
    disp(sprintf('%d \t%s\t\t\t\t\t%4.3f \n',i,micrograph,res));
end
clear('micrograph','i');

%Variables input:
k = input('Enter the number of the micrograph to study: ');
if (k>0)&&(k<59)
    SEM = strcat(FileName(k,:),'.TIF');
    micrograph = FileName(k,:);
    pixsiz = resolution(k);
    Xheight = height(k);
    Ywidth = width(k);
    Workspace = strcat('Workspace',micrograph);
    BlackPixels = strcat('Black',micrograph);
```

```
    Connect = strcat('Connect',micrograph);
    Filtered = strcat('Filtered',micrograph);
    Porous = strcat('Porous',micrograph);
    Porosity = strcat('Porosity',micrograph);
    Xorg = 1;
    Yorg = 1;
else
    disp(sprintf('\n*   *   *   Partial study of a micrograph     *   *
*\n'));
    SEM = input('Enter the micrograph file_name.TIF to study: ','s');
    dot = findstr(SEM,'.');
    micrograph1 = '';
    for i=1:dot-1
        micrograph1 = strcat(micrograph1,SEM(1,i));
    end
    for i=1:LFN
        nb = strfind(FileName(i,:),micrograph1);
        if nb
            lineRes = i;
        end
    end
    pixsiz = resolution(lineRes,:);
    %pixsiz = input('Enter the resolution of one pixel: ');
    disp(sprintf('\n*   *   *   Origin of Study     *   *   *'));
    Xorg = input('Enter the origin X to start the partial study: ');
    maxX = 750-Xorg;
    Yorg = input('Enter the origin Y to start the partial study: ');
    maxY = 1000-Yorg;
    disp(sprintf('\n*   *   *   Size of subsection     *   *   *'));
    disp(sprintf('Choose a height for this image in the following
range: [0 %d]',maxX));
    Xheight = input('Enter the height you want to study: ');
    if (Xheight>maxX)
        disp(sprintf('WARNING: Value off boundaries!\n\t Choose in [0
%d]',maxX));
        Xheight = input('Enter the height you want to study: ');
    end
    disp(sprintf('Choose a widtht for this image in the following
range: [0 %d]',maxY));
    Ywidth = input('Enter the width you want to study: ');
    if (Ywidth>maxY)
        disp(sprintf('WARNING: Value off boundaries!\n\t Choose in [0
%d]',maxY));
        Ywidth = input('Enter the width you want to study: ');
    end
    micrograph = micrograph1;
    Workspace = strcat('Workspace',micrograph);
    BlackPixels = strcat('Black',micrograph);
    Connect = strcat('Connect',micrograph);
    Filtered = strcat('Filtered',micrograph);
    Porous = strcat('Porous',micrograph);
    Porosity = strcat('Porosity',micrograph);
end
clear('micrograph1');
%-------------------------------------------------------------------------
----
imAnalysis(SEM,pixsiz,Xheight,Xorg,Ywidth,Yorg);
```

```
pause;
bkPix(Workspace);
pause;
connectivity(SEM,BlackPixels);
pause;
filtering(SEM,Connect);
pause;
poreAnalysis(SEM,Filtered);
pause;
porosity(SEM,Porous);
pause;

%Working on illustration of results:
load(Porosity);

%-------------------------------------------------------------------------
----
% Define max area size for x-axis:
fid = fopen('maxArea.txt','r');
count = 1;
while ~feof(fid)
    line = fgetl(fid);
    line =fgetl(fid);
    for i=1:length(line)
        extrArea(count,i)=line(i);
    end
    count = count+1;
end
fclose(fid);
clear('line');
clear('fid','i','line','count');

for i=1:length(extrArea(:,1))
    Xmax(i) = str2num(extrArea(i,:));
end
%XmaxAxis = max(Xmax);
XmaxAxis = 550;
%-------------------------------------------------------------------------
----
%1- Summarize pores in an array without any zero values:
% PoresDetail contains every single pores with their respective number
of
% pixels.
k=1;

PoresDetail = [];
for i=1:length(porous(1,:))
    if (porous(1,i)~=0)
        PoresDetail(k)=porous(1,i);
        k = k+1;
    end
end

%2- Cleaning the image from useless pixels:
for i=1:Xpix
    for j=1:Ypix
```

```
        if
(imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0)
            imColor(i,j,1)=1;
            imColor(i,j,2)=1;
            imColor(i,j,3)=1;
        elseif (imColor(i,j,1)==0.49)&&(imColor(i,j,2)==1)&&...
                (imColor(i,j,3)==0.83)
            imColor(i,j,1)=0;
            imColor(i,j,2)=0;
            imColor(i,j,3)=0;
        elseif (imColor(i,j,1)==1)&&(imColor(i,j,2)==0.62)&&...
                (imColor(i,j,3)==0.40)
            imColor(i,j,1)=0;
            imColor(i,j,2)=0;
            imColor(i,j,3)=0;
        end
    end
end
figure(13),imshow(imColor),...
    title('Micrograph after pore counts');
clear('i');
%3- Working on a visualization of pores repartition:
figure(20), plot(PoresDetail),grid on, ...
    title(['Pores repartition in pixels for ',micrograph]),...
    xlabel('Total number of pores'),xlim([0 length(PoresDetail)]),...
    ylabel('Size of pores in pixels'),ylim([0 max(PoresDetail)+100]);
close 20
%% 4- Arrange from min area to max area PoresDetail:
poresCount = sort(PoresDetail); %still in pixel
%%
%5- ploting an histogram with mean values:
nbPores = length(poresCount);
temp(1)=1;
for i=1:nbPores
    if(poresCount(i)<=0.01*poreMax)
        temp(2)=i; %Category of pores less than 1% of Max
    elseif (poresCount(i)<=0.02*poreMax)
        temp(3)=i; %Category of pores less than 2% of Max
    elseif (poresCount(i)<=0.03*poreMax)
        temp(4)=i; %Category of pores less than 3% of Max
    elseif (poresCount(i)<=0.04*poreMax)
        temp(5)=i; %Category of pores less than 4% of Max
    elseif (poresCount(i)<=0.05*poreMax)
        temp(6)=i; %Category of pores less than 5% of Max
    elseif (poresCount(i)<=(poreMax/16))
        temp(7)=i; %Category of pores less than 6.25% of Max
    elseif (poresCount(i)<=(poreMax/8))
        temp(8)=i; %Category of pores less than 12.5% of Max
    elseif (poresCount(i)<=(poreMax/4))
        temp(9)=i; %Category of pores less than 25% of Max
    elseif (poresCount(i)<=(poreMax/2))
        temp(10)=i; %Category of pores less than 50% of Max
    end
end
clear('i','j');
%% poreStats recap data on pores area:
% Line 1: total number of pores, at each step totaled from beginning
```

```
% Line 2: number of pores per category
% Line 3: Average pore size per category
% Line 4: Standard deviation per category; + or - sigma to plot
LT = length(temp(:));
for i=1:LT-1
    poreStats(1,i) = temp(i+1);
    poreStats(2,i) = temp(i+1)-temp(i)+1;
    poreStats(3,i) = mean(poresCount(temp(i):temp(i+1)));
    poreStats(4,i) = std(poresCount(temp(i):temp(i+1)));
end
clear('i');
poreStats(1,LT) = nbPores;
poreStats(2,LT) = nbPores-temp(LT)+1;
poreStats(3,LT) = mean(poresCount(temp(LT):nbPores));
poreStats(4,LT) = std(poresCount(temp(LT):nbPores));
if (sum(poreStats(2,:))==nbPores)
    fprintf(1,'Grand total of pores: %d',nbPores);
else
    fprintf(1,'total of pores in stats: %d',sum(poreStats(2,:)));
end
%% Histogram of the mean values for pores count:
figure(21),
h3 = bar(poreStats(3,:),0.8);
title('Average repartition of pores.'),...
    xlabel('number of pores per category'),...
    ylabel('Area of pores in pixels');
% Write the number of pores above bars:
for i=1:length(poreStats(1,:))
    strgTotal = num2str((poreStats(2,i)));
    text(i,poreStats(3,i)+35,strgTotal,'FontSize',12);
end
clear('h3','strgTotal');
close 21
%% Ploting the number of pores vs size of pores:
% 1- size in µm:
%% Transform values in pixels to values in µm for poresCount and
PoreStats
poresCount = poresCount.*pixArea;
for i=1:LT-1
    poreStats(3,i) = mean(poresCount(temp(i):temp(i+1)));
    poreStats(4,i) = std(poresCount(temp(i):temp(i+1)));
end
clear('i');
poreStats(3,LT) = mean(poresCount(temp(LT):nbPores));
poreStats(4,LT) = std(poresCount(temp(LT):nbPores));
%%
figure(22),
h4 = bar(poreStats(3,:),poreStats(2,:));
title(['Number of pores with respect to the total area: micrograph
',micrograph]),...
    xlabel('average area in in \mum^{2}'),xlim([0 XmaxAxis+1]),...
    ylabel('number of pores (bars)'),ylim([0
max(poreStats(2,:))+5]),...
    grid on;
h1 = gca;
h2 = axes('Position',get(h1,'Position'));
plot(poreStats(3,:),poreStats(2,:),'-ro');
```

```
set(h2,'YAxisLocation','right','Color','none','XTickLabel',[]);
set(h2,'Xlim',get(h1,'Xlim'),'Layer','top');
ylabel('Repartition of number of pores (red curve)');
set(gcf,'PaperPositionMode','auto');
clear('h4','h1','h2');
%
figure(23),
h = bar(poreStats(1,:),poreStats(3,:),0.9); %in µm²
title('Repartition and size of pores.'),...
    xlabel('number of pores per category'),xlim([0 nbPores+1]),...
    ylabel('Average area of pores in \mum^{2}');
colormap hsv;
h1 = gca;
h2 = axes('Position',get(h1,'Position'));
plot(poresCount,'LineWidth',1.25);
set(h2,'YAxisLocation','right','Color','none','XTickLabel',[]);
set(h2,'Xlim',get(h1,'Xlim'),'Layer','top');
text(50,10,'Repartition of pores per size','Rotation',2,'FontSize',9);
ylabel('Exact Repartition of pores area in \mum^{2}');
set(gcf,'PaperPositionMode','auto');
clear('h','h1','h2');
close 23

%%
%-------------------------------------------------------------------------
----
%Summary:
statPorosity = ((sum(PoresDetail))/(Xpix*Ypix))*100;
percentConnect = (connectivity/(Xpix*Ypix))*100;
disp(sprintf('\nSUMMARY:\n\tMicrograph studied: %s,size of the
micrograph: %d x %d.\n\tResolution of one pixel: %1.3f µm x %1.3f
µm.\n\tNumber of pores: %d, for a filtered porosity of %3.2f
%%.\n\tOverall connectivity for this micrograph: %3.2f %%.\n\tCompared
to an apparent porosity at the first evaluation: %3.2f
%%.',SEM,Xpix,Ypix,pixsiz,pixsiz,nbPores,statPorosity,percentConnect,pe
rcent));
%Automatically saving results following the first set:
fid = fopen('ResultsMicrographs.doc','a');
fprintf(fid,'-->   Filtered %s.\n \nSize of the micrograph: %d x
%d.\n\tResolution of one pixel: %1.3f µm x %1.3f µm.\n\tThreshold use
for this study: %2.0f.\n\tNumber of pores: %d, for a filtered porosity
of %3.2f %%.\n\tOverall connectivity for this micrograph: %3.2f
%%.\n\tCompared to an apparent porosity at the first evaluation: %3.2f
%%.\n\n \n
\n',SEM,Xpix,Ypix,pixsiz,pixsiz,holes,nbPores,statPorosity,percentConne
ct,percent);
fclose(fid);
%Saving porosity and connectivity for statistical analysis:
fid = fopen('porosity.txt','a');
fprintf(fid,'%s\n%2.2f\n',micrograph,statPorosity);
fclose(fid);
clear('fid');
fid = fopen('connectivity.txt','a');
fprintf(fid,'%s\n%2.2f\n',micrograph,percentConnect);
fclose(fid);
clear('fid');
```

```
%-------------------------------------------------------------------------
----
%Calculation for the runing time:
t2 = cputime;
totalTime = t2-t1;
hour = floor(totalTime/3600);
rest1 = totalTime - hour*3600;
minute = floor(rest1/60);
seconds = totalTime - (hour*3600 + minute*60);
clear('t1','t2');
%End of Program:
disp(sprintf('*  *   *   End of Program  *   *   *\n Total time to run
the MAIN PROGRAM: %dhr %dmn %1.2fs\n\n',...
    hour,minute,seconds));
clear('totalTime','minute','hour','seconds');
%-------------------------------------------------------------------------
----
%Cleaning useless variables:
clear('t1','t2','samePoreL','samePoreC','begCol','endCol',...
    'SEM','Workspace','BlackPixels','Connect','Filtered',...
    'Porous','Porosity','fid','ans','tolX','tolY','maxX','maxY',...
    'strgSave','count','k','m','map','temp', 'LFN','LT','dot');
%%
%-------------------------------------------------------------------------
----
%Saving the Workspace to be loaded in the next step of analysis:
strgSave = 'Main';
for i=1:length(micrograph)-4
    strgSave = strcat(strgSave,micrograph(1,i));
end
save(strgSave);
```

```
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%
%%%%%%%%          Image Analysis: Contrast and Threshold.
%%%%%%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%
function imAnalysis(micrograph,pixsiz,Xpix,Xorg,Ypix,Yorg)
warning off all;

%%%%%%%%%%%%%%%%%%%%%
%Program Run time
t1 = cputime;
%-----------------------------------------------------------------------
----
%%
%Variables:
Xheight = Xpix + Xorg -1;
Ywidth = Ypix + Yorg -1;
disp(sprintf('\nYour choice is the micrograph: %s \n\twith a resolution
of %1.3f µm x %1.3f µm per pixel\n\tSize of the micrograph studied is
%d x %d\n',micrograph,pixsiz,pixsiz,Xpix,Ypix));
pixArea = pixsiz*pixsiz;% in µm²
totalArea = Xpix*Ypix*pixArea; %in µm²

%-----------------------------------------------------------------------
----
%%
%Every micrographs are 750x1000. Load image:
J = imread(micrograph,'PixelRegion',{[Xorg Xheight] [Yorg Ywidth]});
figure(1),subplot(2,2,1), imshow(J), title('original image');
orgFile = strcat('Original_',micrograph);
imwrite(J,orgFile,'TIFF','Compression','none',...
'WriteMode','overwrite')
%-----------------------------------------------------------------------
----
%%
%Distribution of intensities:
figure(2),subplot(2,2,1), imhist(J), ...
    title('Histogram of intensities: Original');
[counts_Or,X_Or] = imhist(J);% _Or == original.
%Range study on the original image:
maxInt = max(counts_Or);
for i=1:length(counts_Or)
    if (counts_Or(i)> 0.5*maxInt)
        maxRange = X_Or(i);
    end
end
RangeIm_Or = [0 maxRange]; % determined as the last value corresponding
%to half of max intensity.
clear('i');
%-----------------------------------------------------------------------
----
%%
%1- Enhance contrast of image:
```

```
[I2, T] = histeq(J);%Transforms the intensity of the image + gray scale
transf.
figure(1),subplot(2,2,2), imshow(I2), ...
    title('Enhanced contrast image using a classical Histogram
Equalization');

%Another technic: working on tiles in the image rather than the overall
%image:
%2- Exponential equalization
I3 = adapthisteq(J, 'NumTiles', [6 10], 'Distribution', 'exponential');
figure(1),subplot(2,2,3), imshow(I3), ...
    title('Enhanced contrast using an adaptative histogram:
Exponential');
[counts_Exp,X_Exp] = imhist(I3);
%Range study on the modified image with exponential equalization:
maxIntExp = max(counts_Exp);
for i=1:length(counts_Exp)
    if (counts_Exp(i)> 0.5*maxIntExp)
        maxRangeExp = X_Exp(i);
    end
end
RangeIm_Exp = [0 maxRangeExp];
clear('i');
%3- Rayleigh equalization
I4 = adapthisteq(J, 'NumTiles', [6 10], 'Distribution', 'Rayleigh');
figure(1),subplot(2,2,4), imshow(I4), ...
    title('Enhanced contrast using an adaptative histogram: Rayleigh');
[counts_Rayl,X_Rayl] = imhist(I4);
%Range study on the modified image with exponential equalization:
maxIntRayl = max(counts_Rayl); %Takes the maximum value of intensity.
for i=1:length(counts_Rayl)
    if (counts_Rayl(i)> 0.5*maxIntRayl)
        maxRangeRayl = X_Rayl(i);
    end
end
RangeIm_Rayl = [0 maxRangeRayl];
%Distribution of intensities:
figure(2),subplot(2,2,2), imhist(I2), ...
    title('Histogram of intensities: Classic enhanced image');
figure(2),subplot(2,2,3), imhist(I3), ...
    title('Histogram of intensities: Exponential enhanced image');
figure(2),subplot(2,2,4), imhist(I4), ...
    title('Histogram of intensities: Rayleigh enhanced image');
% close the figure:
close 2
%-------------------------------------------------------------------------
----
%%
%Converting to a binary image i.e.illustration of pores vs. surface:

%Calculation of the threshold for pores, using Eponential enhancement:
if maxRangeExp>100
    %round to the max integer inferior to the value.
    holes = floor(2*maxRangeExp/3);
else
    %minimum value in case the Range is too small.
```

```
    holes = 70;
end
%Saving the original in a new variable called: Im
Im = J;
fibers = 1;
for i=1:Xpix
    for j=1:Ypix
        if (J(i,j)>holes)
            Im(i,j)=255;
            fibers = fibers+1; %value in pixels
        end
    end
end
figure(3), imshow(Im), ...
    title('Illustration of pores (Fibers and fines in white)');
%-------------------------------------------------------------------------
----
%Save the picture for record:
strgFile = strcat('Pores_',micrograph);
imwrite(Im,strgFile,'TIFF','Compression','none',...
'WriteMode','overwrite')
%%
%Apparent porosity:
apPores = Xpix*Ypix - fibers; %value in pixels
areaPores = apPores*pixArea; %value in µm²
percent = (apPores/(Xpix*Ypix))*100; %percentage of apparent porosity.
disp(sprintf('Total area of the sample: %9.2fµm².\nTotal area of pores
in the sample: %9.0fµm².\nApparent porosity in the sample: %3.2f
%%',totalArea, areaPores, percent));
%Automatically saving results in a text file:
fid = fopen('ResultsMicrographs.doc','a');
fprintf(fid,'*  *   *   Results for the micrograph: %s. *   *   *\n
\nApparent range for the choice of threshold: [0 %d]\n(The rest of the
image appears in white - value 255 -).\nSample total area: %9.2f
µm².\nPorous total area: %9.0f µm².\nPercentage of pores: %3.2f%%.\n
\nIllustration of pores vs. fibers saved in file: %s.\n \n \n \n',
micrograph,maxRange,totalArea,areaPores,percent,strgFile);
fclose(fid);
%-------------------------------------------------------------------------
----
%Calculation for the runing time:
t2 = cputime;
totalTime = t2-t1;
hour = floor(totalTime/3600);
rest1 = totalTime - hour*3600;
minute = floor(rest1/60);
seconds = totalTime - (hour*3600 + minute*60);
clear('t1','t2');
%End of Program:
disp(sprintf('*  *   *   End of Program  *   *   *\nTotal time to run
image analysis: %dhr %dmn %1.2fs\n\n',hour,minute,seconds));
clear('totalTime','minute','hour','seconds');
%-------------------------------------------------------------------------
----
%Cleaning useless variables:
clear('I2','T','counts_Or','X_Or','maxRange','RangeIm_Or','maxInt',...
```

```
'I3','counts_Exp','X_Exp','maxIntExp','RangeIm_Exp','maxRangeExp',...

'I4','counts_Rayl','X_Rayl','maxIntRayl','RangeIm_Rayl','maxRangeRayl',
...
    'fibers','strgFile','orgFile','apPores','fid','counts',...
    'junk');
%-------------------------------------------------------------------------
----
%Saving the Workspace to be loaded in the next step of analysis:
strgSave = 'Workspace';
for i=1:length(micrograph)-4
    strgSave = strcat(strgSave,micrograph(1,i));
end
save(strgSave);
```

```
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%
%%%%%%%%                      Black Pixels Array
%%%%%%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%

%Creation of an array summarizing the number of black pixels
encountered
%during line scaning of the micrograph.

function bkPix(Workspace)
disp(sprintf('Your current Workspace is: %s \n',Workspace));
%%%%%%%%%%%%%%%%%%%%%
%Program Run time
t1 = cputime;
%-----------------------------------------------------------------------
----

%Variables input:
load(Workspace);
disp(sprintf('The image analyzed is %dx%d\n',Xpix,Ypix));

%Variables for the connectivity test:
tolY = 10;
tolX = 6;
disp(sprintf('\n Criteria for connectivity: %2.0fx%2.0f
pixels\n',tolX,tolY));

%initialization:
k=1;
temp=0;

count = []; col = []; line=[];
for i=1:Xpix
    for j=1:Ypix-1
        if (J(i,j)<=holes)&&(j<=Ypix-1)
            temp = temp + 1;%Count the # holes/pores
            count(k) = temp;%total # of holes
            col(k)=j; %last col of holes
            line(k)=i;%line of the holes
        elseif (J(i,j)>holes)&&(J(i,j+1)<=holes)
            k=k+1;%Skip the non-holes pixels
            temp = 0;
        elseif (J(i,j+1)>holes)&&(j+1==Ypix)
            k=k+1;%End of the line
            temp = 0;
        end
    end
    if (J(i,Ypix)<=holes)%Case of the last element in the line
        temp = temp + 1;% if last element is a hole
        count(k) = temp;
        col(k)=Ypix;
        line(k)=i;
```

```
        k=k+1;%Change of line
        temp = 0;
    end
end
clear('k','temp','i','j');
%%
%Work on Pores array to avoid any cases with 0 pixels in pores:
temp1 = [];endCol = []; line1 = [];
t=1;
for i=1:length(count(1,:))
    if (count(i)~=0)
        temp1(t)=count(i);
        endCol(t)=col(i);
        line1(t)=line(i);
        t=t+1;
    end
end
clear('t','i');
begCol = [];
for j=1:length(temp1)
    begCol(j) = endCol(j) - temp1(j);
end
Bkpix = [temp1; begCol; endCol; line1]; %# of blackpixel per line with
end col.
clear('count','col','line');
clear('temp1','begCol','endCol','line1');
%%
%-------------------------------------------------------------------------
----
%Calculation for the runing time:
t2 = cputime;
totalTime = t2-t1;
hour = floor(totalTime/3600);
rest1 = totalTime - hour*3600;
minute = floor(rest1/60);
seconds = totalTime - (hour*3600 + minute*60);
clear('t1','t2');
%End of Program:
disp(sprintf('*  *   *   End of Program  *   *   *\nTotal time to
determine black pixels: %dhr %dmn %1.2fs\n\n',...
    hour,minute,seconds));
clear('totalTime','minute','hour','seconds');

%-------------------------------------------------------------------------
----
%Saving the Workspace to be loaded in the next step of analysis:
strgSave = 'Black';
for i=1:length(micrograph)-4
    strgSave = strcat(strgSave,micrograph(1,i));
end
save(strgSave);
```

```
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%
%%%%%%%%                 Image analysis in color
%%%%%%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%

function connectivity(micrograph,BlackPixels)

load(BlackPixels);
disp(sprintf('Your current Workspace is: %s. \nCurrent image analyzed:
%s\n',...
    BlackPixels,micrograph));
%%%%%%%%%%%%%%%%%%%%%%
%Program Run time
t1 = cputime;
%-----------------------------------------------------------------------
----

%Working on connectivity pixels: convert image in RGB:
[X,map] = imread(micrograph,'PixelRegion',{[0 Xpix] [0 Ypix]});
imColor = ind2rgb(X,map);
for k=1:length(Bkpix(1,:))
    if Bkpix(1,k)<tolY
        i=Bkpix(4,k);
        for j=Bkpix(2,k):Bkpix(3,k)
            if j>0 %possible errors in black pixels determination.
                imColor(i,j,1)=255;
                imColor(i,j,2)=0;
                imColor(i,j,3)=0;
            else
                for j=1:Bkpix(3,k)
                    imColor(i,j,1)=255;
                    imColor(i,j,2)=0;
                    imColor(i,j,3)=0;
                end
            end
        end
    end
end
% Add surface fibers in white on the image
for i=1:Xpix
    for j=1:Ypix
        if (double(J(i,j))>holes)&&(imColor(i,j,1)~=255)...
                &&(imColor(i,j,2)~=0)&&(imColor(i,j,3)~=0)
            imColor(i,j,1) = 255;
            imColor(i,j,2) = 255;
            imColor(i,j,3) = 255;
        elseif (double(J(i,j))<=holes)&&(imColor(i,j,1)~=255)...
                &&(imColor(i,j,2)~=0)&&(imColor(i,j,3)~=0)
            imColor(i,j,1) = 0;
            imColor(i,j,2) = 0;
            imColor(i,j,3) = 0;
        end
    end
```

```
end
clear('i', 'j');
figure(10),imshow(imColor),...
    title('Illustration of potential connectivity in red pixels');
close 10
%%
%-------------------------------------------------------------------------
----
%Calculation for the runing time:
t2 = cputime;
totalTime = t2-t1;
hour = floor(totalTime/3600);
rest1 = totalTime - hour*3600;
minute = floor(rest1/60);
seconds = totalTime - (hour*3600 + minute*60);
clear('t1','t2');
%End of Program:
disp(sprintf('*  *   *   End of Program  *   *   *\nTotal time to work
on connectivity: %dhr %dmn %1.2fs\n\n',...
    hour,minute,seconds));
clear('totalTime','minute','hour','seconds');
%-------------------------------------------------------------------------
----
%Cleaning useless variables:
clear('X');
%-------------------------------------------------------------------------
----
%Saving the Workspace to be loaded in the next step of analysis:
strgSave = 'Connect';
for i=1:length(micrograph)-4
    strgSave = strcat(strgSave,micrograph(1,i));
end
save(strgSave);
```

```
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%
%%%%%%%%                Filter the image with red pixels
%%%%%%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%

function filtering(micrograph,Connect)

load(Connect);
disp(sprintf('Your current Workspace is: %s. \nCurrent image analyzed:
%s\n',...
    Connect,micrograph));
%%%%%%%%%%%%%%%%%%%%%
%Program Run time
t1 = cputime;
%--------------------------------------------------------------------
----

%Now determine which red pixels are really connectivity:
%=====================================================
for i=2:Xpix-1
    for j=2:Ypix-1
        %Treating alternate colors not verifying connectivity
        %tolerance.
        if
(imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0)...%if
red pixel

&&(imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==255)&&(imColor(i,j+1,3)==
255)...
                &&(imColor(i,j-1,1)==0)&&(imColor(i,j-
1,2)==0)&&(imColor(i,j-1,3)==0)
            imColor(i,j,1)=0;
            imColor(i,j,2)=0;
            imColor(i,j,3)=0;
        elseif
(imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0)...%if
red pixel

&&(imColor(i,j+1,1)==0)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0)..
.
                &&(imColor(i,j-1,1)==255)&&(imColor(i,j-
1,2)==255)&&(imColor(i,j-1,3)==255)
            imColor(i,j,1)=255;
            imColor(i,j,2)=255;
            imColor(i,j,3)=255;
        elseif
(imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0)...%if
red pixel

&&(imColor(i+1,j,1)==0)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0)..
.
                &&(imColor(i-1,j,1)==255)&&(imColor(i-
1,j,2)==255)&&(imColor(i-1,j,3)==255)
```

```
            imColor(i,j,1)=255;
            imColor(i,j,2)=255;
            imColor(i,j,3)=255;
        elseif
(imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0)...%if
red pixel

&&(imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==255)&&(imColor(i+1,j,3)==
255)...
                &&(imColor(i-1,j,1)==0)&&(imColor(i-
1,j,2)==0)&&(imColor(i-1,j,3)==0)
            imColor(i,j,1)=0;
            imColor(i,j,2)=0;
            imColor(i,j,3)=0;
        end
    end
end
clear('i','j');
%Filtering case of 5 red pixels in a row or column:
for i=2:Xpix-5
    for j=2:Ypix-5
        if
((imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0))...
                &&
((imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0))
...
                &&
((imColor(i,j+2,1)==255)&&(imColor(i,j+2,2)==0)&&(imColor(i,j+2,3)==0))
...
                &&
((imColor(i,j+3,1)==255)&&(imColor(i,j+3,2)==0)&&(imColor(i,j+3,3)==0))
...
                &&
((imColor(i,j+4,1)==255)&&(imColor(i,j+4,2)==0)&&(imColor(i,j+4,3)==0))
...
                &&
((imColor(i,j+5,1)==255)&&(imColor(i,j+5,2)==255)&&(imColor(i,j+5,3)==2
55))...
                && ((imColor(i,j-1,1)==255)&&(imColor(i,j-
1,2)==255)&&(imColor(i,j-1,3)==255))
            imColor(i,j,1) = 255;
            imColor(i,j,2) = 255;
            imColor(i,j,3) = 255;
            imColor(i,j+1,1) = 255;
            imColor(i,j+1,2) = 255;
            imColor(i,j+1,3) = 255;
            imColor(i,j+2,1) = 255;
            imColor(i,j+2,2) = 255;
            imColor(i,j+2,3) = 255;
            imColor(i,j+3,1) = 255;
            imColor(i,j+3,2) = 255;
            imColor(i,j+3,3) = 255;
            imColor(i,j+4,1) = 255;
            imColor(i,j+4,2) = 255;
            imColor(i,j+4,3) = 255;
        elseif
((imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0))...
```

```
                &&
((imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0))
...
                &&
((imColor(i,j+2,1)==255)&&(imColor(i,j+2,2)==0)&&(imColor(i,j+2,3)==0))
...
                &&
((imColor(i,j+3,1)==255)&&(imColor(i,j+3,2)==0)&&(imColor(i,j+3,3)==0))
...
                &&
((imColor(i,j+4,1)==255)&&(imColor(i,j+4,2)==0)&&(imColor(i,j+4,3)==0))
...
                &&
((imColor(i,j+5,1)==0)&&(imColor(i,j+5,2)==0)&&(imColor(i,j+5,3)==0))..
.
                && ((imColor(i,j-1,1)==0)&&(imColor(i,j-
1,2)==0)&&(imColor(i,j-1,3)==0))
            imColor(i,j,1) = 0;
            imColor(i,j,2) = 0;
            imColor(i,j,3) = 0;
            imColor(i,j+1,1) = 0;
            imColor(i,j+1,2) = 0;
            imColor(i,j+1,3) = 0;
            imColor(i,j+2,1) = 0;
            imColor(i,j+2,2) = 0;
            imColor(i,j+2,3) = 0;
            imColor(i,j+3,1) = 0;
            imColor(i,j+3,2) = 0;
            imColor(i,j+3,3) = 0;
            imColor(i,j+4,1) = 0;
            imColor(i,j+4,2) = 0;
            imColor(i,j+4,3) = 0;
        end
    end
end
%Filtering the red pixels with a 2x2 square.
%================================================
for i=2:Xpix-1
    for j=2:Ypix-1
        if
(imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0) % (i,j)
red pixel
            if
(imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0)..
.

&&(imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0)
...

&&(imColor(i+1,j+1,1)==255)&&(imColor(i+1,j+1,2)==0)&&(imColor(i+1,j+1,
3)==0)
            %white pixels surrounding:
                if (imColor(i,j-1,1)==255)&&(imColor(i,j-
1,2)==255)&&(imColor(i,j-1,3)==255)...
                    &&(imColor(i-1,j-1,1)==255)&&(imColor(i-1,j-
1,2)==255)&&(imColor(i-1,j-1,3)==255)...
```

```
                    &&(imColor(i-1,j,1)==255)&&(imColor(i-
1,j,2)==255)&&(imColor(i-1,j,3)==255)...
                    &&(imColor(i-1,j+1,1)==255)&&(imColor(i-
1,j+1,2)==255)&&(imColor(i-1,j+1,3)==255)...
                    &&(imColor(i+1,j-1,1)==255)&&(imColor(i+1,j-
1,2)==255)&&(imColor(i+1,j-1,3)==255)
                        imColor(i,j,1) = 255;
                        imColor(i,j,2) = 255;
                        imColor(i,j,3) = 255;
                        imColor(i,j+1,1) = 255;
                        imColor(i,j+1,2) = 255;
                        imColor(i,j+1,3) = 255;
                        imColor(i+1,j,1) = 255;
                        imColor(i+1,j,2) = 255;
                        imColor(i+1,j,3) = 255;
                        imColor(i+1,j+1,1) = 255;
                        imColor(i+1,j+1,2) = 255;
                        imColor(i+1,j+1,3) = 255;
                end
            end
        end
    end
end
clear('i','j');
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%Note: first/end rows/columns are not treated at this point ...
for i=2:Xpix-2
    for j=2:Ypix-2
        %Case of dual red pixels:
        if
(imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0)%if red
pixel (i,j)
            %at position j and j+1 in the line.
            if
(imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0)..
.%if red pixel j+1
                    &&(imColor(i,j-1,1)==255)&&(imColor(i,j-
1,2)==255)&&(imColor(i,j-1,3)==255)...

&&(imColor(i,j+2,1)==255)&&(imColor(i,j+2,2)==255)&&(imColor(i,j+2,3)==
255)
                imColor(i,j,1)=255;
                imColor(i,j,2)=255;
                imColor(i,j,3)=255;
                imColor(i,j+1,1)=255;
                imColor(i,j+1,2)=255;
                imColor(i,j+1,3)=255;
            elseif
(imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0)..
.%if red pixel j+1
                    &&(imColor(i,j-1,1)==0)&&(imColor(i,j-
1,2)==0)&&(imColor(i,j-1,3)==0)...

&&(imColor(i,j+2,1)==0)&&(imColor(i,j+2,2)==0)&&(imColor(i,j+2,3)==0)
                imColor(i,j,1)=0;
                imColor(i,j,2)=0;
                imColor(i,j,3)=0;
```

```
                imColor(i,j+1,1)=0;
                imColor(i,j+1,2)=0;
                imColor(i,j+1,3)=0;
            %red pixel at position i and i+1 in the column.
            elseif
(imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0)..
.%if red pixel i+1
                    &&(imColor(i-1,j,1)==255)&&(imColor(i-
1,j,2)==255)&&(imColor(i-1,j,3)==255)...

&&(imColor(i+2,j,1)==255)&&(imColor(i+2,j,2)==255)&&(imColor(i+2,j,3)==
255)
                imColor(i,j,1)=255;
                imColor(i,j,2)=255;
                imColor(i,j,3)=255;
                imColor(i+1,j,1)=255;
                imColor(i+1,j,2)=255;
                imColor(i+1,j,3)=255;
            elseif
(imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0)..
.%if red pixel i+1
                    &&(imColor(i-1,j,1)==0)&&(imColor(i-
1,j,2)==0)&&(imColor(i-1,j,3)==0)...

&&(imColor(i+2,j,1)==0)&&(imColor(i+2,j,2)==0)&&(imColor(i+2,j,3)==0)
                imColor(i,j,1)=0;
                imColor(i,j,2)=0;
                imColor(i,j,3)=0;
                imColor(i+1,j,1)=0;
                imColor(i+1,j,2)=0;
                imColor(i+1,j,3)=0;
            end
        end
    end
end
for i=2:Xpix-3
    for j=2:Ypix-3
        %Case of three red pixels:
        if
(imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0)%if red
pixel
            %at position j, j+1 and j+2 in the line.
            if
(imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0)..
.%if red pixel j+1

&&(imColor(i,j+2,1)==255)&&(imColor(i,j+2,2)==0)&&(imColor(i,j+2,3)==0)
...% if red pixel j+2
                    &&(imColor(i,j-1,1)==255)&&(imColor(i,j-
1,2)==255)&&(imColor(i,j-1,3)==255)...

&&(imColor(i,j+3,1)==255)&&(imColor(i,j+3,2)==255)&&(imColor(i,j+3,3)==
255)
                imColor(i,j,1)=255;
                imColor(i,j,2)=255;
                imColor(i,j,3)=255;
                imColor(i,j+1,1)=255;
```

```
                imColor(i,j+1,2)=255;
                imColor(i,j+1,3)=255;
                imColor(i,j+2,1)=255;
                imColor(i,j+2,2)=255;
                imColor(i,j+2,3)=255;
            elseif
(imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0)..
.%if red pixel j+1

&&(imColor(i,j+2,1)==255)&&(imColor(i,j+2,2)==0)&&(imColor(i,j+2,3)==0)
...%if red pixel j+2
                    &&(imColor(i,j-1,1)==0)&&(imColor(i,j-
1,2)==0)&&(imColor(i,j-1,3)==0)...

&&(imColor(i,j+3,1)==0)&&(imColor(i,j+3,2)==0)&&(imColor(i,j+3,3)==0)
                imColor(i,j,1)=0;
                imColor(i,j,2)=0;
                imColor(i,j,3)=0;
                imColor(i,j+1,1)=0;
                imColor(i,j+1,2)=0;
                imColor(i,j+1,3)=0;
                imColor(i,j+2,1)=0;
                imColor(i,j+2,2)=0;
                imColor(i,j+2,3)=0;
            %red pixel at position i, i+1 and i+2 in the column.
            elseif
(imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0)..
.%if red pixel i+1

&&(imColor(i+2,j,1)==255)&&(imColor(i+2,j,2)==0)&&(imColor(i+2,j,3)==0)
...%if red pixel i+2
                    &&(imColor(i-1,j,1)==255)&&(imColor(i-
1,j,2)==255)&&(imColor(i-1,j,3)==255)...

&&(imColor(i+3,j,1)==255)&&(imColor(i+3,j,2)==255)&&(imColor(i+3,j,3)==
255)
                imColor(i,j,1)=255;
                imColor(i,j,2)=255;
                imColor(i,j,3)=255;
                imColor(i+1,j,1)=255;
                imColor(i+1,j,2)=255;
                imColor(i+1,j,3)=255;
                imColor(i+2,j,1)=255;
                imColor(i+2,j,2)=255;
                imColor(i+2,j,3)=255;
            elseif
(imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0)..
.%if red pixel i+1

&&(imColor(i+2,j,1)==255)&&(imColor(i+2,j,2)==0)&&(imColor(i+2,j,3)==0)
...%if red pixel i+2
                    &&(imColor(i-1,j,1)==0)&&(imColor(i-
1,j,2)==0)&&(imColor(i-1,j,3)==0)...

&&(imColor(i+3,j,1)==0)&&(imColor(i+3,j,2)==0)&&(imColor(i+3,j,3)==0)
                imColor(i,j,1)=0;
                imColor(i,j,2)=0;
```

```
                imColor(i,j,3)=0;
                imColor(i+1,j,1)=0;
                imColor(i+1,j,2)=0;
                imColor(i+1,j,3)=0;
                imColor(i+2,j,1)=0;
                imColor(i+2,j,2)=0;
                imColor(i+2,j,3)=0;
            end
        end
    end
end
clear('i','j');
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%Case of single red pixel checking:
for i=2:Xpix-1
    for j=2:Ypix-1
        if
(imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0)...%if
red pixel

&&(imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==255)&&(imColor(i,j+1,3)==
255)...

&&(imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==255)&&(imColor(i+1,j,3)==
255)...
                &&(imColor(i,j-1,1)==255)&&(imColor(i,j-
1,2)==255)&&(imColor(i,j-1,3)==255)...
                &&(imColor(i-1,j,1)==255)&&(imColor(i-
1,j,2)==255)&&(imColor(i-1,j,3)==255)
            imColor(i,j,1)=255;
            imColor(i,j,2)=255;
            imColor(i,j,3)=255;
        elseif
(imColor(i,j,1)==255)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0)...%if
red pixel

&&(imColor(i,j+1,1)==0)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0)..
.

&&(imColor(i+1,j,1)==0)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0)..
.
                &&(imColor(i,j-1,1)==0)&&(imColor(i,j-
1,2)==0)&&(imColor(i,j-1,3)==0)...
                &&(imColor(i-1,j,1)==0)&&(imColor(i-
1,j,2)==0)&&(imColor(i-1,j,3)==0)
            imColor(i,j,1)=0;
            imColor(i,j,2)=0;
            imColor(i,j,3)=0;
        end
    end
end
clear('i','j');

%-------------------------------------------------------------------------
----
%Case of dual white pixels surrounded by black pixels:
for i=2:Xpix-2
```

```
    for j=2:Ypix-2
        if
((imColor(i,j,1)==255)&&(imColor(i,j,2)==255)&&(imColor(i,j,3)==255))..
.
                &&
((imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==255)&&(imColor(i,j+1,3)==2
55))...
                && ((imColor(i,j-1,1)==0)&&(imColor(i,j-
1,2)==0)&&(imColor(i,j-1,3)==0))...
                &&
((imColor(i,j+2,1)==0)&&(imColor(i,j+2,2)==0)&&(imColor(i,j+2,3)==0))..
.
                && ((imColor(i-1,j,1)==0)&&(imColor(i-
1,j,2)==0)&&(imColor(i-1,j,3)==0))...
                && ((imColor(i-1,j+1,1)==0)&&(imColor(i-
1,j+1,2)==0)&&(imColor(i-1,j+1,3)==0))...
                &&
((imColor(i+1,j,1)==0)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0))..
.
                &&
((imColor(i+1,j+1,1)==0)&&(imColor(i+1,j+1,2)==0)&&(imColor(i+1,j+1,3)=
=0))
            imColor(i,j,1)=0;
            imColor(i,j,2)=0;
            imColor(i,j,3)=0;
            imColor(i,j+1,1)=0;
            imColor(i,j+1,2)=0;
            imColor(i,j+1,3)=0;
        elseif
((imColor(i,j,1)==255)&&(imColor(i,j,2)==255)&&(imColor(i,j,3)==255))..
.
                &&
((imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==255)&&(imColor(i+1,j,3)==2
55))...
                && ((imColor(i-1,j,1)==0)&&(imColor(i-
1,j,2)==0)&&(imColor(i-1,j,3)==0))...
                && ((imColor(i,j-1,1)==0)&&(imColor(i,j-
1,2)==0)&&(imColor(i,j-1,3)==0))...
                && ((imColor(i+1,j-1,1)==0)&&(imColor(i+1,j-
1,2)==0)&&(imColor(i+1,j-1,3)==0))...
                &&
((imColor(i,j+1,1)==0)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0))..
.
                &&
((imColor(i+1,j+1,1)==0)&&(imColor(i+1,j+1,2)==0)&&(imColor(i+1,j+1,3)=
=0))...
                &&
((imColor(i+2,j,1)==0)&&(imColor(i+2,j,2)==0)&&(imColor(i+2,j,3)==0))
            imColor(i,j,1)=0;
            imColor(i,j,2)=0;
            imColor(i,j,3)=0;
            imColor(i,j+1,1)=0;
            imColor(i,j+1,2)=0;
            imColor(i,j+1,3)=0;
        end
    end
end
```

```
%-------------------------------------------------------------------------
----
%Case of single white pixel surrounded by black pixels:
for i=2:Xpix-1
    for j=2:Ypix-1
        if
((imColor(i,j,1)==255)&&(imColor(i,j,2)==255)&&(imColor(i,j,3)==255))..
.
                && ((imColor(i-1,j,1)==0)&&(imColor(i-
1,j,2)==0)&&(imColor(i-1,j,3)==0))...
                &&
((imColor(i+1,j,1)==0)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0))..
.
                && ((imColor(i,j-1,1)==0)&&(imColor(i,j-
1,2)==0)&&(imColor(i,j-1,3)==0))...
                &&
((imColor(i,j+1,1)==0)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0))
            imColor(i,j,1)=0;
            imColor(i,j,2)=0;
            imColor(i,j,3)=0;
        end
    end
end
clear('i','j');
figure(11),imshow(imColor),...
    title('Connectivity after filtering red pixels on fibers');
%%
%-------------------------------------------------------------------------
----
%Calculation for the runing time:
t2 = cputime;
totalTime = t2-t1;
hour = floor(totalTime/3600);
rest1 = totalTime - hour*3600;
minute = floor(rest1/60);
seconds = totalTime - (hour*3600 + minute*60);
clear('t1','t2');
%End of Program:
disp(sprintf('*  *   *   End of Program     *    *   *\n Total time to
work on filtering: %dhr %dmin %1.2fs\n\n',...
    hour,minute,seconds));
%-------------------------------------------------------------------------
----
%Saving the Workspace to be loaded in the next step of analysis:
strgSave = 'Filtered';
for i=1:length(micrograph)-4
    strgSave = strcat(strgSave,micrograph(1,i));
end
save(strgSave);
```

```
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%%
%%%%%%%%%                    Computing Pores total area
%%%%%%%%%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%%

function poreAnalysis(micrograph,Filtered)

load(Filtered);
disp(sprintf('Your current Workspace is: %s. \nCurrent image analyzed:
%s\n',...
    Filtered,micrograph));
%%%%%%%%%%%%%%%%%%%%%
%Program Run time
t1 = cputime;
%-----------------------------------------------------------------------
----

% 1-
%Create an array as done for Bkpix to recollect every black pixels in
%imColor:
%initialization:
k=1;
temp=0;

count = []; col = []; line = [];
for i=1:Xpix
    for j=1:Ypix-1
        if
((imColor(i,j,1)==0)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0))...
                &&(j<=Ypix-1)
            temp = temp + 1;%Count the # black pixel/pores
            count(k) = temp;%total # of holes
            col(k)=j; %last col of holes
            line(k)=i;%line of the holes
        elseif (imColor(i,j,1)==255)...

&&((imColor(i,j+1,1)==0)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0))
            k=k+1;%Skip the non-holes pixels only at last non black
pixel.
            temp = 0;
        elseif (imColor(i,j+1,1)==255)&&(j+1==Ypix)
            k=k+1;%End of the line
            temp = 0;
        end
    end
    %treating the case of j=Ypix, last element in the column.
    if
(imColor(i,Ypix,1)==0)&&(imColor(i,Ypix,2)==0)&&(imColor(i,Ypix,3)==0)%
Case of the last element in the line
        temp = temp + 1;% if last element is a hole
        count(k) = temp;
        col(k)=Ypix;
        line(k)=i;
```

```
        k=k+1;%Change of line
        temp = 0;
    end
end
%Work on Pores array to avoid any cases with 0 pixels in pores:
%=============================================================
temp1 = [];endCol = []; line1 = [];
t=1;
for i=1:length(count(1,:))
    if (count(i)~=0)
        temp1(t)=count(i);
        endCol(t)=col(i);
        line1(t)=line(i);
        t=t+1;
    end
end
clear('t','i');
begCol = [];
for j=1:length(temp1)
    begCol(j) = endCol(j) - temp1(j)+1;
end
porous1 = [temp1; begCol; endCol; line1]; %# of blackpixel per line.
clear('count','col','line');
clear('temp1','begCol','endCol','line1');
%Check if any begCol(k)<0, if so, replace by 1.
for j=1:length(porous1(2,:))
    if (porous1(2,j)<=0)
        porous1(2,j)=1;
    end
end
clear('j');
%
%Checking if pixel at begCol-1 and endCol+1 of porous1 are black:
%That would mean that some pixels are not counted as part of pores.
for i=1:length(porous1(1,:))
    if ((porous1(2,i)-1)>=2)
        if (imColor(porous1(4,i),porous1(2,i)-1,1)==0)
            disp(sprintf('value for porous1 at k = %d, [%d %d
%d],coordinates: (%d,%d)',...
                i,imColor(porous1(4,i),porous1(2,i)-1,1),...
                imColor(porous1(4,i),porous1(2,i)-1,2),...
                imColor(porous1(4,i),porous1(2,i)-1,3),...
                porous1(4,i),porous1(2,i)-1));
        end
    end
    if ((porous1(3,i)+1)<=Ypix-1)
        if (imColor(porous1(4,i),porous1(3,i)+1,1)==0)
            disp(sprintf('value for porous1 at k = %d, [%d %d
%d],coordinates: (%d,%d)\n',...
                i,imColor(porous1(4,i),porous1(3,i)+1,1),...
                imColor(porous1(4,i),porous1(3,i)+1,2),...
                imColor(porous1(4,i),porous1(3,i)+1,3),...
                porous1(4,i),porous1(3,i)+1));
        end
    end
end
%%
```

```
%-------------------------------------------------------------------------
----
%Calculation for the runing time:
t2 = cputime;
totalTime = t2-t1;
hour = floor(totalTime/3600);
rest1 = totalTime - hour*3600;
minute = floor(rest1/60);
seconds = totalTime - (hour*3600 + minute*60);
clear('t1','t2');
%End of Program:
disp(sprintf('*  *   *   End of Program  *   *   *\n Total time to work
on pore analysis: %dhr %dmn %1.2fs\n\n',...
    hour,minute,seconds));
clear('totalTime','minute','hour','seconds');
%-------------------------------------------------------------------------
----
%Saving the Workspace to be loaded in the next step of analysis:
strgSave = 'Porous';
for i=1:length(micrograph)-4
    strgSave = strcat(strgSave,micrograph(1,i));
end
save(strgSave);
```

```
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%%
%%%%%%%%%                  Porosity analysis:
%%%%%%%%%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%%

function porosity(micrograph,Porous)

load(Porous);
disp(sprintf('Your current Workspace is: %s. \nCurrent image analyzed:
%s\n',...
    Porous,micrograph));
%%%%%%%%%%%%%%%%%%%%%
%Program Run time
t1 = cputime;
%-------------------------------------------------------------------------
----

%================
%Pores Total Area
%================
nextPoreL = 0;
nextPoreC = 0;
samePoreL = 0;
samePoreC = 0;
porous= zeros(1,length(Bkpix(1,:)));
k = 1;
connectivity = 0;
i=2;
j=2;
while i<Xpix+1,
    while j<Ypix+1,
        if
(imColor(i,j,1)==0)&&(imColor(i,j,2)==0)&&(imColor(i,j,3)==0)
            imColor(i,j,1)=0.49;
            imColor(i,j,2)=1;
            imColor(i,j,3)=0.83;%Put the current black pixel in
aquamarine.
            %determine the position of current black pixels in porous1:
            t=1;
            while t<=length(porous1(4,:)), %search first line i
              if (porous1(4,t)<i)
                  t = t+1; %skip as long as t~=i, current line.
              else
                  if (porous1(3,t)<j)%search then with respect to
column number.
                      t = t+1;
                  else
                      if (porous1(2,t)<=j)
                          begCol = porous1(2,t);
                          endCol = porous1(3,t);
                          if (porous1(1,t)>=0)
                              porous(1,k) = porous(1,k) + porous1(1,t);
%add values to current pore
```

```
                                porous1(1,t) = -1; %assign -1 to get rid
of this value in the checking.
                                t=length(porous1(4,:))+1; %exit the loop
on t!
                            else
                                %If porous1 is already scanned, value -1,
just
                                %exit the loop:
                                t=length(porous1(4,:))+1;
                            end
                        else
                            disp(sprintf('begCol and endCol are not
initialized at (%d,%d), corresponding to values in porous1: line = %d,
begining = %d and finishing = %d column.',...

i,j,porous1(4,t),porous1(2,t),porous1(3,t)));
                            %If case doesn't match, just
                            %exit the loop:
                            t=length(porous1(4,:))+1;
                            %define begCol and endCol for safety?
                            begCol=j;
                            endCol=j+1;
                        end
                    end
                end
            end
            clear('t');

            %-------------------------------------------------------------
----
            %Treat the case where black pixels in (i-1,j) are in the
same
            %pore:
            %-------------------------------------------------------------
----
            if (i>1)
                if ((imColor(i-1,j,1)==0)&&(imColor(i-
1,j,2)==0)&&(imColor(i-1,j,3)==0))
                    t=1;
                    while t<=length(porous1(4,:)), %search
corresponding line i-1
                        if (porous1(4,t)<i-1)
                            t = t+1; %skip as long as t~=i=-1, current
line.
                        else
                            if (porous1(3,t)<j)%search then with respect
to column number.
                                t = t+1;
                            else
                                if (porous1(2,t)<=j)
                                    begColUp = porous1(2,t);
                                    endColUp = porous1(3,t);
                                    if (porous1(1,t)>=0)
                                        porous(1,k) = porous(1,k) +
porous1(1,t); %add values to current pore
                                        porous1(1,t) = -1; %assign -1 to
get rid of this value in the checking.
```

```
                                    t=length(porous1(4,:))+1; %exit
the loop on t!
                                else
                                    %If porous1 is already scanned,
value -1, just
                                    %exit the loop:
                                    t=length(porous1(4,:))+1;
                                end
                            else
                                %If case doesn't match, just
                                %exit the loop:
                                t=length(porous1(4,:))+1;
                            end
                        end
                    end
                end
                clear('t');
                for m=begColUp:endColUp
                    %put the pixels of PREVIOUS line in imColor in
                    %aquamarine
                    imColor(i-1,m,1)=0.49;
                    imColor(i-1,m,2)=1;
                    imColor(i-1,m,3)=0.83;
                end
                %Define a return point at this stage for the
current Pore:
                if (nextPoreL>i-2)
                    nextPoreL = i-2;
                    nextPoreC = j;
                elseif ((nextPoreL==i-2)&&(nextPoreC>j))
                    nextPoreL = i-2;
                    nextPoreC = j;
                end
                %keep studying the line:
                i=i;
                if (j<=Ypix-1)
                    j=j+1;
                else
                    j = 2;
                end
                clear('begColUp','endColUp');
            end %end of the case previous line is part of the
current pore.
        end

        %--------------------------------------
        %case of connectivity, red pixel found:
        %--------------------------------------
        % 1- next line, same column:
        if
((imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0))
            connectivity = connectivity + 1;
            %Connectivity pixel in copper to mark it as counted.
            imColor(i+1,j,1)=1;
            imColor(i+1,j,2)=0.62;
            imColor(i+1,j,3)=0.4;
            %keep studying the line:
```

```
                i=i;
                j=j+1; %go to next pixel in the line.
            end
            % 2- same line, next column:
            if
((imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0))
                connectivity = connectivity + 1;
                %Connectivity pixel in copper to mark it as counted.
                imColor(i+1,j,1)=1;
                imColor(i+1,j,2)=0.62;
                imColor(i+1,j,3)=0.4;
                %end of the line for the current pore:
                %Mark the return point to study next pore:
                if (nextPoreL>i)
                    nextPoreL = i;
                    nextPoreC = j+2;
                elseif ((nextPoreL==i)&&(nextPoreC>j+1))
                    nextPoreL = i;
                    nextPoreC = j+2;
                end
            end

            %-------------------------------------------------------
            %Put a marker in case pixel at (i+1,j) is black also
            %i.e. pixel below is in the same pore:
            %-------------------------------------------------------
            if
(imColor(i+1,j,1)==0)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0)...

||(imColor(i+1,j,1)==0.49)&&(imColor(i+1,j,2)==1)&&(imColor(i+1,j,3)==0
.83)
                %Define return point for a pixel below in the same
pore:
                %Two cases: either this pixel is black or aquamarine.
                if ((samePoreL==0)&&(samePoreC==0))
                    if (samePoreL>i+1)
                        samePoreL = i+1;
                        samePoreC = j;
                    elseif ((samePoreL==i)&&(samePoreC>j))
                        samePoreL = i+1;
                        samePoreC = j;
                    end
                end
            end

            %-------------------------------------------------------------
            %Switching line at the end of current line of black pixels:
            %-------------------------------------------------------------
            if ((j==endCol+1)&&(i+1==samePoreL))
                %Define a return point if there's none defined already,
                %Before switching line.
                if (nextPoreL>i)
                    nextPoreL = i;
                    nextPoreC = j+1;
                elseif ((nextPoreL==i)&&(nextPoreC>j))
                    nextPoreL = i;
```

```
                    nextPoreC = j+1;
                end
                %go to next line if i is not the last line in picture:
                if (i<=Xpix-1)
                    i=i+1; %i now takes the value of next line!
                    j = samePoreC;
                    %Assign values to samePore at zero now they're
used:
                    samePoreL = 0;
                    samePoreC = 0;
                    %determine j to start at the right column:
                    t=1;
                    while t<=length(porous1(4,:)), %search
corresponding line i
                      if (porous1(4,t)<i)
                          t = t+1; %skip as long as t~=i+1, current
line.
                      else
                          if (porous1(3,t)<j)%search then with respect
to column number.
                              t = t+1;
                          else
                              if (porous1(2,t)<=j)
                                  begColDown = porous1(2,t);
                                  endColDown = porous1(3,t);
                                  if (porous1(1,t)>=0)
                                      porous(1,k) = porous(1,k) +
porous1(1,t); %add values to current pore
                                      porous1(1,t) = -1; %assign -1 to
get rid of this value in the checking.
                                      t=length(porous1(4,:))+1; %exit
the loop on t!
                                  else
                                      %If porous1 is already scanned,
value -1, just
                                      %exit the loop:
                                      t=length(porous1(4,:))+1;
                                  end
                              else
                                  %If case doesn't match, just
                                  %exit the loop:
                                  t=length(porous1(4,:))+1;
                              end
                          end
                      end
                    end
                    clear('t');
                    %force values of i and j to be treated after the
switched
                    %line in the if case.
                    i=i;
                    j=begColDown; %Force the entire line to be scanned.
                    clear('begColDown','endColDown');
                else
                    i = Xpix+1;
                end
            end
```

```
            %----------------------------------------
            %Stoping cases concerning the current pore:
            %----------------------------------------
            %both pixels (i,j+1) and (i+1,j) are not black: k = k+1, go
to
            %nextPore line and Col for i and j:
            %NOTE: consider just white and red pixels here,
            %Blue pixels are not a stoping case in the current pore.
            if ((samePoreL~=0)&&(samePoreC~=0))
                %go back to count pixels in the same pore if not
alreday
                %done:
                i = samePoreL;
                j = samePoreC;
                samePoreL = 0;
                samePoreC = 0;
            elseif ((j<Ypix-1)&&(i<=Xpix-1))
                if
((imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==255)&&(imColor(i,j+1,3)==2
55)...

&&(imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==255)&&(imColor(i+1,j,3)==
255))...

|||((imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0
)...

&&(imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0)
)...

|||((imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==255)&&(imColor(i,j+1,3)=
=255)...

&&(imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==0)&&(imColor(i+1,j,3)==0)
)...

|||((imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0
)...

&&(imColor(i+1,j,1)==255)&&(imColor(i+1,j,2)==255)&&(imColor(i+1,j,3)==
255))
                    %Then go to next Pore:
                    if ((nextPoreL~=0)&&(nextPoreC~=0))
                        i = nextPoreL; % line of the return point
                        j = nextPoreC; %Col of the return point
                        k = k+1; %Count black pixels for the next pore.
                        %clean return point:
                        nextPoreL = 0;
                        nextPoreC = 0;
                    else
                        k = k+1;
                        i = i;
                        j = j+2;
                    end
                    if ((i<=Xpix-1)&&(j<=Ypix-1))
```

```
                        %no need to consider this connectivity at last
line
                        %or last column
                        if
((imColor(i+1,j+1,1)==0)&&(imColor(i+1,j+1,2)==0)&&(imColor(i+1,j+1,3)=
=0))
                            %Consider this case as pseudo connectivity
between
                            %two pores:
                            connectivity = connectivity + 1;
                        end
                    end
                else
                    if (i>=Xpix)
                        if (j>=Ypix-1)
                            %Exit loop
                            i = Xpix+1;
                            j = Ypix+1;
                        end
                    end
                    if (j>=Ypix)
                        if (i>=Xpix-1)
                            %Exit loop
                            i = Xpix+1;
                            j = Ypix+1;
                        end
                    end
                end
            end
        end % end for if pixel (i,j) == black
        %^^^^^^^
        %NOTE:
        %if current pixel is aquamarine, red, or white, keep scanning
the line:
        if ((imColor(i,j,1)~=0)&&(imColor(i,j,1)~=1))
            i=i;
            j=j+1;
        end
        %If current pixel is copper -> part of connectivity:
        if (j<=Ypix)
            if (imColor(i,j,1)==1)
                if (j<=Ypix-1)
                    if
((imColor(i,j+1,1)==255)&&(imColor(i,j+1,2)==0)&&(imColor(i,j+1,3)==0))
                        connectivity = connectivity + 1;
                        imColor(i+1,j,1)=1;
                        imColor(i+1,j,2)=0.62;
                        imColor(i+1,j,3)=0.4;
                        %Skip the copper pixel to check for more
connectivity
                        i=i;
                        j=j+1;
                    else
                        i=i;
                        j=j+1;
                    end
                elseif (i<=Xpix-1)
```

```
                    connectivity = connectivity + 1;
                    i=i+1;
                    j=2;
                else
                    connectivity = connectivity + 1;
                    i=Xpix+1;
                    j=Ypix+1;
                end
            end
        end
        %^^^^^^
        %Consider boundary cases:
        if (i==0)
            i = 2;
        elseif (i==Xpix)
            if (j>=Ypix-1)
                %Exit loop:
                i = Xpix+1;
                j = Ypix+1;
            else
                %Go to the line above to check any missing pixels:
                i = Xpix-1;
            end
        end
        if (j==0)
            j = 1;
        elseif (j>=Ypix-1)
            if (i>=Xpix-1)
                %Exit loop:
                i = Xpix+1;
                j = Ypix+1;
            else
                %Go the column before checking any missing pixels:
                j = Ypix-1;
            end
        end
        %-------------------------------------------------------------
----
        %Treat the case where i and j  are at the boundaries: Exit Loop
        %-------------------------------------------------------------
----
        if (j+1>=Ypix)
            if(i+1>=Xpix)
                i=Xpix+1;
                j=Ypix+1;
            else
                i=i+1;
                j=2;
            end
        end
    end %end for while j.
end% end for while i.
figure(12),imshow(imColor),...
    title(['Pores detailed, results of pores count in blue.
',micrograph]);
clear('i','j');
close 12
```

```
%%
% Save major values for stat study:
poreMax = max(porous);
% Maximum area in µm²
maxArea = poreMax * pixArea;
% Saving the max area value in µm x µm
fid = fopen('maxArea.txt','a');
fprintf(fid,'%s\n%2.2f\n',micrograph,maxArea);
fclose(fid);
clear('fid');
%%
%-------------------------------------------------------------------------
----
%Calculation for the runing time:
t2 = cputime;
totalTime = t2-t1;
hour = floor(totalTime/3600);
rest1 = totalTime - hour*3600;
minute = floor(rest1/60);
seconds = totalTime - (hour*3600 + minute*60);
clear('t1','t2');
%End of Program:
disp(sprintf('*  *   *   End of Program  *   *   *\n Total time to work
on porosity: %dhr %dmn %1.2fs\n\n',...
    hour,minute,seconds));
clear('totalTime','minute','hour','seconds');
%-------------------------------------------------------------------------
----
%Cleaning useless variables:
clear('nextPoreL','nextPoreC','samePoreL','samePoreC',...
    'Bkpix','ans');
%-------------------------------------------------------------------------
----
%Saving the Workspace to be loaded in the next step of analysis:
strgSave = 'Porosity';
for i=1:length(micrograph)-4
    strgSave = strcat(strgSave,micrograph(1,i));
end
save(strgSave);
```

```
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%
%%%%%%%%                      Statistic application.
%%%%%%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%

%The goal of this file is to analyze results given in terms of porosity
and
%connectivity in a statistical way. For example, a series of ten
%micrographs is taken from the sample called 89, we'll take the average
and
%standard deviation concerning 89_SEM_001 to 89_SEM_010.
%First thing to do is to scan the saved text files containing
respectively
%the name of each micrographs, the percentages of porosity and the
%percentages of connectivity.

clear, close all;
warning off all;

%%
%Read the file containing the porosity in percentage and the file
%containing the micrographs detail to switch values per name.
%Create an array containing name of the micrographs and another
containing
%the percentage of porosity:
fid = fopen('detailMicrographs.txt','r');
count = 1;
while ~feof(fid)
   j=1;
   line = fgetl(fid);
   if isempty(line)||(line(1)=='F'), continue, end
   k = strfind(line, 'TIF');
   for i=1:k-2
        FileName(count,j)=line(i);
        j = j+1;
   end
   clear('k','i');
   count = count + 1;
end
fclose(fid);
clear('fid','j','line','count');
%
%Array containing all percentages with associated micrographs:
fid = fopen('porosity.txt','r');
count = 1;
line = fgetl(fid);
k = strfind(line, 'SEM');
if (k~=0)
        for i=1:k+2
            SavedName(count,i)=line(i);
        end
    end
while ~feof(fid)
```

```
    count = count+1;
    line = fgetl(fid);
    line = fgetl(fid);
    k = strfind(line, 'SEM');
    if (k~=0)
        for i=1:k+2
            SavedName(count,i)=line(i);
        end
    end
end
fclose(fid);
clear('k','fid','count','i');

fid = fopen('porosity.txt','r');
count = 1;
while ~feof(fid)
    line = fgetl(fid);
    line =fgetl(fid);
    for i=1:length(line)
        percent(count,i)=line(i);
    end
    count = count+1;
end
fclose(fid);
clear('line');
clear('fid','i','line','count');

fid = fopen('connectivity.txt','r');
count = 1;
while ~feof(fid)
    line = fgetl(fid);
    line =fgetl(fid);
    for i=1:length(line)
        connect(count,i)=line(i);
    end
    count = count+1;
end
fclose(fid);
clear('line');
clear('fid','i','line','count');
%%
%To create a statistical repartition of results, we're going to scan
here
%the file containing the official name of micrographs and summarize the
%different "file_SEM" name we have:
p=1;
list(p,1:6)=' ';
for i=1:length(FileName(:,1))
    temp='';
    k = strfind(FileName(i,:), 'SEM');
    if (k~=0)
        for j=1:k+2
            temp = strcat(temp,FileName(i,j));
        end
    end
    for i=1:length(temp)
```

```
        tempList(i) = list(p,i);
    end
    if ~isequal(tempList,temp)
        for j=1:length(temp)
            list(p+1,j)=temp(j);
        end
        p = p+1;
    end
end
clear('p','tempList','temp','k','FileName');
for i=1:length(list)-1
    for j=1:length(list(1,:))
        recordList(i,j) = list(i+1,j);
    end
end
clear('list','i','j');

%tip: to look at the content of a saved file.txt:
%type myfile.txt e.g. type porosity.txt
%%
%Now put together all percentages that correspond to a same file_SEM in
%order to get an average for this particular handsheet containing
several
%samples. The idea is to compare with the official list 'recordList' to
the
%scanned files 'SavedName' and then reorganize the percentages.
p=1;
for i=1:length(recordList(:,1))
    for j=1:length(SavedName(:,1))
        if isequal(recordList(i,:),SavedName(j,:))
            for t=1:length(SavedName(j,:))
                Stat1(p,t)= SavedName(j,t); %sort names in the scanned
files
            end
            for q=1:length(percent(j,:))
                Stat2(p,q) = percent(j,q);
            end
            for k=1:length(connect(j,:))
                Stat3(p,k) = connect(j,k);
            end
            p = p+1;
        end
    end
end
clear('i','j','p','q','t','k');
%Take the average of percentages for each sample 'file_SEM':
p=1; q=1;
for i=1:length(Stat1(:,1))-1
    if isequal(Stat1(i,:),Stat1(i+1,:))
        sumPercent(p,q) = str2double(Stat2(i,:));
        q = q+1;
    else
        listName(p,:)=Stat1(i,:);
        sumPercent(p,q) = str2double(Stat2(i,:));
        p = p+1;
        q = 1;
    end
```

```
end
if isequal(Stat1(length(Stat1(:,1))-1,:),Stat1(length(Stat1(:,1)),:))
    listName(p,:)=Stat1(length(Stat1(:,1)),:);
    sumPercent(p,q) = str2double(Stat2(length(Stat1(:,1)),:));
else
    listName(p,:)=Stat1(length(Stat1(:,1)),:);
    sumPercent(p,1) = str2double(Stat2(length(Stat1(:,1)),:));
end
clear('p','q','i');

%Take the average of connectivities for each sample 'file_SEM':
p=1; q=1;
for i=1:length(Stat1(:,1))-1
    if isequal(Stat1(i,:),Stat1(i+1,:))
        sumConnect(p,q) = str2double(Stat3(i,:));
        q = q+1;
    else
        sumConnect(p,q) = str2double(Stat3(i,:));
        p = p+1;
        q = 1;
    end
end
if isequal(Stat1(length(Stat1(:,1))-1,:),Stat1(length(Stat1(:,1)),:))
    sumConnect(p,q) = str2double(Stat3(length(Stat1(:,1)),:));
else
    sumConnect(p,1) = str2double(Stat3(length(Stat1(:,1)),:));
end
clear('p','q','i');
%%
%Summarizing and saving results:

%Automatically saving results in a text file:
fid = fopen('Percentages.doc','a');
for i=1:length(sumPercent(:,1))
    k=0;
    %summary(1,i)= listName(i,:);
    for j=1:length(sumPercent(1,:))
        if sumPercent(i,j)>0
            k = k+1;
        end
    end
    Avg = mean(sumPercent(i,1:k));
    summary(2,i) = Avg;
    StdDev = std(sumPercent(i,1:k));
    summary(3,i) = StdDev;
    %
    p=0;
    for j=1:length(sumConnect(1,:))
        if sumConnect(i,j)>0
            p = p+1;
        end
    end
    Avg1 = mean(sumConnect(i,1:p));
    summary(4,i) = Avg1;
    StdDev1 = std(sumConnect(i,1:p));
    summary(5,i) = StdDev1;
```

```
    fprintf(fid,'*  *   *   Results for the micrograph: %s. *   *   *\n
\nAverage porosity: %3.2f%%.\n\tStandard Deviation: %3.2f.\nAverage
connectivity for this sample: %3.2f%%.\n\tStandard Deviation:
%3.2f.\n\n',listName(i,:),Avg,StdDev,Avg1,StdDev1);
end
fclose(fid);
clear('fid','i','j','k','ans');
%%
% Integrate Standard deviations to the values of percentage porosity
and
% connectivity:
ResSTD = [];
for i=1:length(summary(1,:))
    ResSTD(1,i) = summary(2,i) - summary(3,i);
    ResSTD(2,i) = summary(2,i) + summary(3,i);
    ResSTD(3,i) = summary(4,i) - summary(5,i);
    ResSTD(4,i) = summary(4,i) + summary(5,i);
    ResSTD(5,i) = i;
end
xminP = 0;
xmaxP = max(ResSTD(5,:));
ymin1P = min(ResSTD(1,:));
ymin2P = min(ResSTD(2,:));
yminP = min(ymin1P,ymin2P);
ymax1P = max(ResSTD(1,:));
ymax2P = max(ResSTD(2,:));
ymaxP = max(ymax1P,ymax2P);
%%
%Histogram of the mean values for pores count:
figure(1),
subplot(2,1,1),h3 = bar(summary(2,:),0.8,'r');
hold on
for j=1:length(ResSTD(1,:))
    X1 = [ResSTD(5,j) ResSTD(5,j)];
    Y1 = [ResSTD(1,j) ResSTD(2,j)];
    plot(X1,Y1,'LineWidth',1.50);
        xlim([xminP xmaxP+1]);
        ylim([yminP ymaxP+1]);
    hold on
end
hold off
title('Average porous percentages.'),...
    xlabel('cases'),...
    ylabel('Percentage of porosity');
xminC = 0;
xmaxC = max(ResSTD(5,:));
ymin1C = min(ResSTD(3,:));
ymin2C = min(ResSTD(4,:));
yminC = min(ymin1C,ymin2C);
ymax1C = max(ResSTD(3,:));
ymax2C = max(ResSTD(4,:));
ymaxC = max(ymax1C,ymax2C);
subplot(2,1,2),h5 = bar(summary(4,:),0.7,'r');
hold on
for j=1:length(ResSTD(1,:))
    X1 = [ResSTD(5,j) ResSTD(5,j)];
    Y1 = [ResSTD(3,j) ResSTD(4,j)];
```

```
    plot(X1,Y1,'LineWidth',1.50);
    xlim([xminC xmaxC+1]);
    ylim([yminC ymaxC+1]);
    hold on
end
hold off
title('Average connectivity percentages.'),...
    xlabel('cases'),...
    ylabel('Percentage of connectivity');
```

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